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Assessment of Techniques for 'In Situ' Repair of Flexible Membrane Liners

Matrecon, Inc., Alameda, CA

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Experimental studies relating to preparation of HDPE seams made with a heat gun indicate the importance of surface preparation in achieving adhesion between the two pieces of liner being seamed. Cleanliness and fresh surfaces are necessary. Compatibility tests of six different potential plugging materials (five epoxies and one cement) in five simulated test media show the importance of the medium on the ability of the material to set and with long-term integrity of the cured materials.

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ASSESSMENT OF TECHNIQUES FOR IN SITU REPAIR OF FLEXIBLE MEMBRANE LINERS

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EPA Purchase Order No. 5C2520 NAST

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FOREWORD

Today's rapidly developing and changing technologies and industrial products and practices frequently carry with them the increased generation of solid and hazardous waste. These materials, if improperly dealt with, can threaten both public health and the environment. Abandoned waste sites and acidental releases of toxic and hazardous substances to the environment also have important environmental and public health implications. The Hazardous Waste Engineering Reseach Laboratory assists in providing an authoritative and defensible engineering basis for assessing and solving these problems. Its products support the policies, programs, and regulations of the EPA; the permitting and other responsibilities of state and local governments; and the needs of both large and small businesses in handling their wastes responsibly and economically.

The following report presents the results of an investigation into the feasibility of repairing damaged flexible membrane liners (FMLs) in situ while they are in service in landfills, surface impoundments, and waste piles. In this investigation, both patching and plugging of holes, seam openings, and other damage to in-service FMLs were considered. The results of searching the open literature for information on actual repairs of FMLs are presented. The characteristics of various FMLs that are currently in service, current methods used in seaming FMLs, and current test methods used in quality control of seaming operations are discussed. The results of determining the chemical compatibility of HDPE seams made with a heat gun and of six potential plugging materials with five different test media, including acidic, alkaline, and organic media, are reported.

For further information, please contact the Land Pollution Control Division of the Hazardous Waste Engineering Research Laboratory.

Thomas R. Hauser, Director
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ABSTRACT

The feasibility of using either patching or plugging to make *in situ* repairs of damaged areas in in-service flexible membrane liners (FMLs) was investigated. The open literature, including data from the liner industry, was surveyed for information on repairing in-service FMLs, without success. Literature on seaming, adhesives, and polymer adhesion was reviewed, as was information on the type of polymeric FMLs in service, the type of seaming and patching methods currently used, and methods of testing and assessing seams to minimize the possibility of seam failure. The potential use of specific materials to plug holes in in-service FMLs was also reviewed. Experimental work was conducted on the compatibility of seams of an HDPE FML and of six potential plugging materials in five different test media that simulated various types of waste streams.

The investigation indicated that there is no current technology that can be used to repair leaks and other damage in FMLs that are in service below wastes. Applying the basic criteria used in assessing and testing liners and seams in FMLs that are being installed, it appears highly questionable that conditions required for preparation of adequate seams and permanent repairs can be met with FMLs exposed below wastes. Liners exposed to the weather only can be repaired if the proper conditions of cleanliness and dryness are met. Repairing with formed-in-place plugs holds some promise for short-term use; however, the compatibility of the plugging material with the waste liquid should be assessed.

Experimental studies relating to preparation of HDPE seams made with a heat gun indicate the importance of surface preparation in achieving adhesion between the two pieces of liner being seamed. Cleanliness and fresh surfaces are necessary. Compatibility tests of six different potential plugging materials (five epoxies and one cement) in five simulated test media show the importance of the medium on the ability of the material to set and with long-term integrity of the cured materials.

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SECTION 1

INTRODUCTION

The use of flexible membrane liners (FMLs) in liner systems for waste storage and disposal facilities has been increasing over the past decade. The principal basis for this application of FMLs is their low permeability to liquid flow and their ability to minimize the escape of waste constituents and subsequent contamination of surface or groundwater. While these liners can be quite effective in reducing environmental contamination, the potentially low permeability of the liner can be compromised by membrane defects during manufacture, by seam defects, by excessive swelling, or by damage occurring accidentally during installation or as a result of wear and tear during service.

This report discusses the technical requirements and conditions for repairing an emplaced FML and techniques used in temporary and possible permanent in situ repairs of FMLs at various times during installation and service. The results of searching the existing technical literature on seaming polymeric sheeting and the results of discussing the general industry practice with technical experts are presented, along with laboratory results using different repair techniques. Laboratory test procedures used in assessing repair methods are described and test results are presented.

In view of the desirability of being able to repair damaged in-place (FMLs) without having to remove large quantities of waste from above them, the original project was undertaken to assess the feasibility of making permanent in situ repairs of in-place membrane and admix liner systems.

This report was prepared under EPA Purchase Order No. P.O. 5C2520 NAST, which is a follow-on to EPA Contract 68-03-1714 with EarthTech Research, Inc., on which Matrecon, Inc. was a subcontractor.

SECTION 2

SUMMARY OF WORK

The open technical literature was searched for information on the repair of FMLs. The search included literature on adhesion, adhesives, and various methods of seaming coated fabrics, membranes, and FMLs. However, outside of work either performed by Matrecon under previous EPA contracts or observed by Matrecon personnel, little information was found relating to the repair of FMLs in the field or under simulated field conditions.

The general concept of repairing liners was investigated with particular reference to the potential problems that might occur during the different phases in the life of a liner. Also investigated was the concept of temporary versus permanent repair.

Criteria for repairing liners in the field were scrutinized. It was felt that the basic requirement for a permanent repair should be that it meet the same criteria that the liner and seam must meet at the time of liner installation. If a patch is used, its adhesion to the liner should equal that of the original seams. On the other hand, if a formed-in-place plug is used to repair a hole in a liner, chemical adhesion to the liner may not be required if a sufficiently high mechanical bond is formed between the liner and the plug.

A meeting of the consultants on the original project was held to discuss the feasibility of repairing damaged liners in the field, particularly liners that were in service below ground level and under wet conditions. The consensus of these consultants was that work on this project should concentrate on investigating the repair of damage to high-density polyethylene (HDPE) FMLs and possibly the use of plugs for repairing damage to FMLs below the surface.

Experimental work was carried out to:

1. Assess methods of seaming polyethylene with a Leister gun and by spin-welding.

2. Assess the compatibility of polyethylene seams in contact with a series of five test media. These test media included deionized water, 10% HCl, 10% NaOH, a 5% solution of mineral oil in xylene,

and a mixed solvent containing equal volumes of chloroform, methyl ethyl ketone, and cyclohexane.

3. Assess the compatibility of six potential plugging materials in contact with the same series of five test media. These potential plugging materials were all hardenable liquid compositions and included five epoxies and one hydraulic cement. In one study, the ability of the plugging materials to harden in the presence of the test media and, if they did harden, whether they maintained their properties in continued exposure were determined. A second study was performed in which these plugging materials were cured in air and then immersed in the test media.

SECTION 3

CONCLUSIONS AND RECOMMENDATIONS

CONCLUSIONS

The long-term integrity of seams made in a repair operation is questionable. Repairing a damaged FML by patching the liner with pieces of the same type of membrane material of essentially the same composition can only be performed if the liner is clean and dry and has a fresh surface. These conditions are prerequisites for achieving good adhesion between materials in general and are very difficult to achieve in the environment of waste disposal facilities, except perhaps on slopes and in areas of the FML not exposed to the weather or to the waste.

The difficulties involved in making quality repairs that meet the criteria imposed on a newly installed liner make it necessary to emphasize quality control and quality assurance during the construction of a disposal facility and the installation of a liner so as to minimize the possibility of liner failure. It appears that, if there is inaccessible damage below the waste in an in-place liner, the damage is nonrepairable and it may be necessary to close the disposal unit.

Plugging holes in a liner with hardenable liquids, such as the epoxies, may be feasible (1) if good mechanical bonds can be made between the plug and the liner, (2) if the liquid is deliverable to the location of the damage, and (3) if the plugging material is compatible with the waste liquid during cure and subsequent exposure.

Because of uncertainty about the adequacy of liner repairs in situ below solid waste, double liner systems are essential to ensure protection of the groundwater system.

RECOMMENDATIONS

At the present state of lining technology and given the difficulties involved with repairing in-service FMLs and the potential closure of an impoundment unit once there is damage to the liner, it is recommended that large impoundment units be avoided and that smaller units be used which can be monitored individually. Small units can be cleaned out or closed without moving massive quantities of waste, thus reducing the risk of groundwater contamination.

Due to the lack of information in the open technical literature on the durability of FML seams, failures of in-service FMLs, and the repair of in-service FMLs, field verification studies should be emphasized

to assess the types of failures that occur, for example, whether seams break open or whether damage is done by puncturing a liner. An effort should be made to locate and assess field repairs of liners made during installation and performance testing.

Increased emphasis should be placed on the development of seaming techniques, including the development of new techniques. Also, increased emphasis should be placed on seam testing to ensure that seams with long-term integrity and durability are prepared and accepted during installation.

Samples of seams should be included in the compatibility testing of the FMLs during the liner selection and design process. In addition to the quality assurance testing of seams performed during construction on seam samples cut from the actual liner, similar samples should be exposed and routinely tested during service as part of coupon testing to monitor the quality of the seams.

SECTION 4

POLYMERIC MEMBRANES AND METHODS OF SEAMING AND TESTING SEAMS

INTRODUCTION

A wide range of different FMLs is in service at the present time. These FMLs are based on a variety of polymers and may be either unreinforced or fabric reinforced and either crosslinked or thermoplastic. Eventually, some of these liners may fail. In this section the pertinent polymers are described, and the properties that relate to potential methods of repairing liners made of the respective polymers are discussed. As seams appear to be one of the major sources of failure of FMLs in the field, methods used in seaming and joining polymeric membranes to form large sheets of FMLs are also described. The various potential methods of repairing damaged FMLs that may be used for different materials and different types of service are described. Methods of assessing the integrity of seams, patches, and other repairs are also described.

FLEXIBLE POLYMERIC MEMBRANE LINERS IN SERVICE

Types and Compositions of FMLs

Polymers that have been used in the manufacture of lining materials include rubbers and plastics. These polymers differ in basic chemical composition and structure, solubility characteristics, chemical resistance, and the mode in which they deteriorate and ultimately fail. The polymeric compositions used in the manufacture of FMLs can be classified into four general types:

- Rubbers (elastomers) that are generally crosslinked.
- Thermoplastic elastomers that are not crosslinked but may contain some crystallinity, which exists at normal service temperatures.
- Thermoplastics that are generally not crosslinked.
- Plastics that are thermoplastic and have a relatively high crystalline content, such as the polyethylenes.

Table 1 lists the types of polymers that are, or have been, used in the manufacture of FMLs. Table 1 also presents information on FMLs by the respective polymer type. The most frequently used polymers have been PVC, CSPE, CPE, IIR, EPDM, CR, and HDPE. Commercial polymeric membranes for liners range in thickness from 0.5 mm (20 mils) to 3.0 mm (120 mils), with

most in the 0.75-mm (30-mil) to 1.5-mm (60-mil) range. Calendering can be used to produce membranes that are unreinforced or reinforced with a fabric; these membranes include FMLs based on both rubbers and thermoplastics. Liners based on semicrystalline polymers, such as polyethylene, are produced by extrusion and are unreinforced.

TABLE 1. POLYMERIC FLEXIBLE MEMBRANE LINERS

Polymer	Abbreviation	Type of compound[a]	Thickness, mm	Fabric reinforced
Butyl rubber	IIR	XL	0.9-2.3	Yes, no
Chlorinated polyethylene	CPE	TP	0.5-1.0	Yes, no
Chlorosulfonated polyethylene	CSPE	TP,XL	0.6-1.0	Yes, no
Elasticized polyolefin	ELPO	TP-CX	0.6	No
Elasticized polyvinyl chloride	PVC-E	TP	0.8-0.9	Yes
Epichlorohydrin rubber	ECO	XL	1.67	Yes, no
Ethylene propylene rubber	EPDM	XL,TP	0.5-1.6	Yes, no
Neoprene	CR	XL	0.5-1.8	Yes, no
Nitrile rubber	NBR	XL	0.8	Yes
Polybutylene	PB	TP-CX	0.8	No
Polyester elastomer	PEL	TP-CX	0.5-0.8	Yes, no
Polyethylene, Low-density	LDPE	TP-CX	0.3-0.8	No
Linear low-density	LLDPE	TP-CX	0.5-2.0	No
High-density	HDPE	TP-CX	0.5-3.1	No
Polyvinyl chloride	PVC	TP	0.5-0.9	Yes, no
Polyvinyl chloride, oil-resistant	PVC-OR	TP	0.8	No

[a]TP = Thermoplastic; TP-CX = Semicrystalline thermoplastic; XL = Crosslinked.

Most polymeric FMLs have been based on a single polymer, but blends of two or more polymers have been developed and used in liners, e.g., blends of CPE and PVC. Also, different grades of a given type of polymer have been used, e.g., polyethylene. All polymeric compositions are formulated with auxiliary ingredients which serve different functions in the compounds.

The basic compositions of the different types of compounds are shown in Table 2. The crosslinked compositions are usually the most complex because they contain a crosslinking system. Thermoplastics, except for CSPE compounds, contain no curative. Although supplied as thermoplastic, CSPE liners contain crosslinking agents that allow the polymer to crosslink during service. Thus, though a CSPE sheeting may be thermoplastic when placed, it is compounded to crosslink somewhat after it has been in service. The crosslinking increases chemical resistance but creates problems for making repairs of the sheeting.

TABLE 2. BASIC COMPOSITIONS OF POLYMERIC FML LINER COMPOUNDS

Component	Composition of compound type, parts by weight		
	Cross-linked	Thermo-plastic	Semicrys-talline
Polymer or blend	100	100	100
Oil or plasticizer	5-40	5-55	0-10
Fillers:			
Carbon black	5-40	5-40	2-5
Inorganics	5-40	5-40	...
Antidegradants	1-2	1-2	1
Crosslinking system:			
Inorganic system	5-9	[a]	...
Sulfur system	5-9	...	...

[a]An inorganic curing system that crosslinks over time is incorporated in CSPE liner compounds.

Semicrystalline FMLs, e.g., polyethylene, have the simplest composition and generally consist of the polymer, a small amount of carbon black for ultraviolet protection, antidegradants, and possibly minor amounts of processing aids. Such FMLs are thermoplastic and retain their ability to melt and to be welded for a considerable length of time in most environmental conditions.

The composition of an FML can change during service. These changes can reduce repairability, even when the changes are slight and do not affect overall performance of the liner. Low molecular weight fractions in the original polymer or blend can be lost. Auxiliary components in a composition can be affected during service when the membrane is either immersed in liquids or exposed to the weather. The oils and plasticizers are potentially extractable and, in some cases, biodegradable. Anti-degradants can also be extracted during liner service. In addition, processing aids in the compound may adversely affect adhesion when seams and repairs are made. Matching the composition of an exposed liner with that of a patching material is important in achieving a good bond between the aged material and a new unexposed patching or repair material.

Most of the polymeric membrane liners that have been or are being manufactured are thermoplastic. They will probably be the types of FMLs that will be encountered in the field for repair. Some polymers, such as CPE and CSPE, are more chemically resistant when crosslinked. Nevertheless, they have been generally used as thermoplastics in liners because it was, and still is, easier to obtain reliable seams and to make repairs in the field with thermoplastic sheeting. Thermoplastic FMLs can be heat-sealed or seamed with a solvent or bodied solvent. FMLs based on semicrystalline polymers, such as HDPE, are generally seamed by thermal welding or fusion methods. (Note: semicrystalline polymers are also thermoplastic.) Crosslinked FMLs must be seamed or repaired with adhesives which generally require treated and clean surfaces (see Section 5).

Membranes of all but the semicrystalline-type compositions are manufactured and available with fabric reinforcement which increases thermal stability and generally increases tensile modulus and strength. Fabric constructions, such as square woven or leno weave, vary in threads per inch from 6 x 6 ends per inch (epi) to more than 20 x 20 epi. As the thread count increases, the area between the threads that allows contact between the plies is reduced. Adhesion between plies is dependent upon the area and good contact and flow of the polymeric layers between the threads during manufacture. Fabric-to-rubber adhesion in most FMLs is minimal. During tear testing of the FML this lack of adhesion between the fabric and rubber allows the fabric threads to break away from the polymeric coating and gather together and thus impart good tear resistance to the construction. Good initial ply adhesion and its retention are important to prevent delamination of the FML during service and loss of overall integrity. Loss of ply adhesion in fabric-reinforced membranes has resulted in a type of failure that is impossible to repair without exposing and replacing large areas of the membrane.

More detailed information on individual polymers and liners is presented in the EPA Technical Resource Document on liners (Matrecon, 1983).

Membrane Manufacture

The three basic methods used in the manufacture of FMLs for liner use are calendering, extrusion, and spread or knife coating. Calendering is

used in forming both unreinforced and fabric-reinforced sheeting, whereas extrusion is only used in making unreinforced sheeting. Spread coating is used for making fabric-reinforced sheeting in wnich the fabric is comparatively tight, i.e., the number of threads per inch is greater than 20.

Calendering has been the most common method of forming FML sheeting. In this process, heated rubber or thermoplastic compounds are passed between the heated rolls to form a sheet of predetermined thickness. Both three- and four-roll calenders are used. Unreinforced sheeting is usually of single-ply construction; however, some manufacturers ply two or more unreinforced layers of compound to minimize the occurrence of pinholes through the sheet.

Reinforcing fabric can be placed between the plies of the polymeric compound to produce a reinforced membrane. In this case, sufficient coating compound is placed on both sides of the fabric so that pinholes are not generated between the fabric and the outer surfaces. Also, sufficient compound is applied to "strike through" the open weave of the fabric and achieve direct contact of the rubber on both sides of the fabric. Open fabrics or scrim of nylon, polyester, and polypropylene are used for fabric reinforcement. The thread count usually ranges from 6 x 6 to 10 x 10 epi, but some are 20 x 20 epi. For most reinforced thermoplastic membranes, the fabric is treated after weaving to tack the yarns in place but no adhesive is applied. Consequently, there is almost no adhesion between the fabric and the polymeric coating compound.

Fabrics used in vulcanized elastomeric FMLs are usually treated with an adhesive coating which chemically reacts with the membrane coating during the curing cycle to produce adhesion to the polymer compound. Different coating formulations are used, depending on the end use. The vulcanization of this type of FML is generally performed on large rolls in large autoclaves.

The extrusion technique is used to manufacture semicrystalline FMLs, such as polyethylene. For some extrusion methods, a tube of film is produced and slit to form a sheet. In other extrusion operations, thin sheets are extruded directly. The width of extruded FMLs varies from 6 to 33 feet. The wide sheets are brought to the field in rolls. The narrow sheets, e.g., those 6-ft wide, are fabricated in a factory into rolls which are then transported to the field site.

Spread coating is performed only on fabrics having a high thread count. In this process, the coating compound is applied as a viscous "dough" made of a high concentration of the compound dispersed in a solvent. The fabric is first passed over a spreader bar to remove wrinkles and creases and then passed beneath a stationary blade which spreads the compound and controls the thickness of the polymer coating. The solvent is evaporated by drawing the coated fabric through a heated chamber and the solvent is recovered. Upon removal from the heated areas, the sheeting is cooled and rolled. The coating compound is thermoplastic, and the adhesion between the fabric and the coating is high.

Testing of Flexible Polymeric Membranes

Because of the wide range of compositions and constructions used in the manufacture of polymeric membrane liners and the fact that they originated in the rubber, plastics, and textile industries, an extensive number of tests are used to evaluate these materials. Table 3 presents a listing of analytical, physical, and environmental exposure and aging test methods that can be used for thermoplastic, crosslinked, semicrystalline, and fabric-reinforced sheetings. Most of the test methods are ASTM. However, two methods are from Federal Test Method Standard 101C, and three were developed during work on EPA projects and are presented in the Technical Resource Document on liners (Matrecon, 1983).

METHODS USED IN SEAMING FMLS

The nature of both the base polymer and additives affects the choice of bonding or seaming methods that can be used as the polymers differ greatly in their chemistry and structure and these differences have the predominant effect on seaming method choice. For example, only thermoplastic liners are heat fusible to a liquid state and, thus, amenable to a heat seaming method. In addition, because noncrystalline thermoplastics are soluble in some solvents, they can also be joined with either solvents or bodied solvents.

During the seaming operation in the installation of a new liner, rigid quality assurance/quality control is being instituted to assure the adequacy of the seams. Depending on the function of a repair and its required durability, a quality control program similar to that used to monitor seaming operations should be imposed.

The methods of seaming and joining FMLs that have been used in the fabrication of panels in the factory and installation in the field, and which were considered for possible use in repairing damaged FMLs in service, are summarized in Table 4. Note that the table was derived from information on new, unexposed liner materials (Haxo, 1983, p 98). The individual methods are discussed briefly below. All of the methods, however, require dryness and cleanliness of the surfaces to be joined, conditions that are often very difficult, if not impossible, to meet in the field when attempting repairs.

Thermal Methods

A variety of thermal seaming methods are applicable to thermoplastic FMLs which soften, melt, and flow at higher temperatures. The thermoplastics are listed in Table 1. Thermal seaming methods include heat gun, fusion welding, hot-wedge, heat sealing, dielectric heat, and combinations. Factory seams of elastomeric FMLs are vulcanized.

Heat Gun--

Seaming with a heat gun has been used for all types of thermoplastic membranes under both factory and field conditions, including repair of

TABLE 3. APPROPRIATE OR APPLICABLE TEST METHODS FOR FLEXIBLE MEMBRANE LINERS

Property	Membrane Liner Without Fabric Reinforcement			Fabric reinforced
	Thermoplastic	Crosslinked	Semicrystalline	
Analytical properties				
Volatiles	MTM-1[a]	MTM-1[a]	MTM-1[a]	MTM-1[a] (on selvage and reinforced sheeting)
Extractables	MTM-2[a]	MTM-2[a]	MTM-2[a]	MTM-2[a] (on selvage and reinforced sheeting)
Ash	ASTM D297, Section 34	ASTM D297, Section 34	ASTM D297, Section 34	ASTM D297, Section 34 (on selvage)
Specific gravity	ASTM D792, Mtd A	ASTM D297, Section 15	ASTM D792, Mtd A	ASTM D792, Mtd A (on selvage)
Thermal analysis:				
Differential scanning calorimetry (DSC)	na	na	yes	na
Thermogravimetry (TGA)	yes	yes	yes	yes
Physical properties				
Thickness - total	ASTM D638	ASTM D412	ASTM D638	ASTM D751, Section 6
Coating over fabric	na	na	na	Optical Method
Tensile properties	ASTM D882, ASTM D638	ASTM D412	ASTM D638 (mod)	ASTM D751, Mtd A and B (ASTM D638 on selvage)
Tear resistance	ASTM D1004 (mod)	ASTM D624, Die C	ASTM D1004	ASTM D751, Tongue Mtd (8 in. x 8-in. specimens)
Modulus of elasticity	na	na	ASTM D882, Mtd A	na
Hardness	ASTM D2240 Duro A or D	ASTM D2240 Duro A or D	ASTM D2240 Duro A or D	ASTM D2240 Duro A or D (selvage only)
Puncture resistance	FTMS 101C, Mtd 2065	FTMS 101C, Mtd 2065	FTMS 101C, Mtd 2065	FTMS 101C, Mtd 2031 and 2065
Hydrostatic resistance	na	na	ASTM D751, Mtd A	ASTM D751, Mtd A
Seam strength:				
In shear	ASTM D882, Mtd A (mod)	ASTM D882, Mtd A (mod)	ASTM D882, Mtd A (mod)	ASTM D751, Grab Method (mod)
In peel	ASTM D413, Mach Mtd Type 1 (mod)	ASTM D413, Mach Mtd Type 1 (mod)	ASTM D413, Mach Mtd Type 1 (mod)	ASTM D413, Mach Mtd Type 1 (mod)
Ply adhesion	na	na	na	ASTM D413, Mach Mtd Type 1 ASTM D751, Sections 39-42
Environmental and aging effects				
Ozone-cracking	ASTM D1149	ASTM D1149	na	ASTM D1149
Environmental stress-cracking	na	na	ASTM D1693	na
Low-temperature testing	ASTM D1790	ASTM D746	ASTM D1790 ASTM D746	ASTM D2136
Tensile properties at elevated temperature	ASTM D638 (mod)	ASTM D412 (mod)	ASTM D638 (mod)	ASTM D751, Mtd B (mod)
Dimensional stability	ASTM D1204	ASTM D1204	ASTM D1204	ASTM D1204
Air-oven aging	ASTM D573 (mod)	ASTM D573 (mod)	ASTM D573 (mod)	ASTM D573 (mod)
Water vapor transmission	ASTM E96, Mtd BW	ASTM E96, Mtd BW	ASTM E96, Mtd BW	ASTM E96, Mtd BW
Water absorption	ASTM D570	ASTM D471	ASTM D570	ASTM D570
Immersion in standard liquids	ASTM D471, D543	ASTM D471	ASTM D543	ASTM D471, D543
Immersion in waste liquids	EPA 9090	EPA 9090	EPA 9090	EPA 9090
Soil burial	ASTM D3083	ASTM D3083	ASTM D3083	ASTM D3083
Outdoor exposure	ASTM D4364	ASTM D4364	ASTM D4364	ASTM D4364
Tub test	Haxo et al, 1985	Haxo et al, 1985	Haxo et al, 1985	Haxo et al, 1985

[a]Matrecon, 1983.
na = Not applicable.

TABLE 4. BONDING METHODS USED FOR SEAMING POLYMERIC MEMBRANE LINERS IN THE FACTORY AND IN THE FIELD

Polymer	Type of compound[a]	Place used	Thermal methods					Solvent methods		Adhesives		Tape[b]
			Heat gun	Extrusion welding	Hot wedge	Heat-sealed	Dielectric	Solvent welding	Bodied-solvents	Contact adhesives	Vulcanizing cements	
Butyl rubber	XL	Factory	...	...	...	...	...	...	...	X	X	...
		Field	...	...	...	...	...	...	...	X	X	X
Chlorinated polyethylene	TP	Factory	X	...	X	X	X	X	X	X	...	...
		Field	X	...	X	X	...	X	X	X	...	X
Chlorosulfonated polyethylene	TP	Factory	X	...	X	X	X	X	X	X	...	...
		Field	X	...	X	X	...	X	X	X	...	X
Elasticized polyolefin	TP	Factory	X	...	X	X	...	...	...	X	...	...
		Field	X	...	X	X	...	...	...	X	...	...
Ethylene propylene rubber	XL	Factory	...	...	...	...	...	...	...	X	X	...
		Field	...	...	...	...	...	...	...	X	X	X
Low-density polyethylene	TP/CX	Factory	X	...	...	X	...	...	...	X	...	...
		Field	X	...	...	X	...	...	...	X	...	X
High-density polyethylene	TP/CX	Factory	...	X	X	X	...	...	...	...	...	...
		Field	...	X	X	X	...	...	...	...	...	...
Neoprene (polychloroprene)	XL	Factory	...	...	...	...	...	...	...	X	...	...
		Field	...	...	...	...	...	...	...	X	...	...
Polyvinyl chloride	TP	Factory	X	...	X	X	X	X	X	X	X	...
		Field	X	...	X	X	...	X	X	X	X	X

[a]XL = Crosslinked or vulcanized; TP = thermoplastic; CX = semicrystalline.

[b]Tape was used in some early installations, but is not adequate for hazardous waste disposal facilities.

unexposed liners. In this method, high temperature air or an inert gas, such as nitrogen, is directed between two sheets to melt the surfaces to be joined. The two pieces are then forced together with pressure and allowed to cool to form a lap seam (Haxo, 1983, p 100; Skeist, 1965, p 156).

The major advantage of the heat gun method is its broad range of application to many thermoplastic materials. The two disadvantages are the great care required to obtain uniform, reproducible seams and the tendency of the polymer surface to react with oxygen (i.e., to oxidize) or otherwise decompose thermally during the seaming process and thus result in a poor bond. This method also requires that the surfaces to be joined be clean and free of moisture, dust, oil, and all solvents. This requirement poses problems when seaming in the field, particularly when seaming FMLs that have been exposed to waste streams and to the weather (see Section 6).

Extrusion Welding--

Some seaming of HDPE liners is being performed in the field with a variety of proprietary and specially designed seaming equipment. Several such devices extrude molten HDPE of the same composition as the liner either between the sheetings being seamed or at the edge of one sheet to form a bead or fillet. Also, seaming equipment based upon heat guns has been devised in which coiled plastic welding rods are incorporated. The rod is fed to the seam area to form a welded seam. In the first welding procedure, a jet of hot air is injected into the overlap area to blow away debris and heat the area to be welded. Directly following the hot air, a ribbon of molten polymeric compound identical to that of the sheet is injected into the overlap through an extruder nozzle. A roller moving behind the extruder nozzle presses the overlap together so the sheets will be fused by the extruded ribbon. The result can be a homogeneous weld that is immediately load bearing. Welding can be carried out in ambient temperatures between 5°C and 35°C (Haxo, 1983, p 100).

Extrusion and fusion seaming methods have great advantages for field use as continuous seams of extended length can be laid down. The plastic mass constituting the seam is supplemented by the extruded bead or welding rod, improving seam strength and consistency. These methods, as with all other methods, require careful preparation of the surface to be bonded (e.g., drying and buffing, and proper adjustment of temperatures at the surfaces of the layers to joined) to assure melt and flow of the polymeric compound. Correct temperature control is essential to assure flow across the interface where the two sheets are joined.

Hot-Wedge--

The hot-wedge method (Neidhart, 1979) consists of a hot electrically-heated element in the shape of a blade or V-shaped wedge that is passed between the two sheets to be sealed. Contacting the two sheets to be seamed together, the heated element melts and smears the two surfaces,

causing fresh material to come to the surface. Immediately following the melting, roller pressure brings the molten surfaces together to form a homogeneous bond.

The hot-wedge method is particularly suited for the thicker [greater than 0.76 mm (30 mils)] LLDPE and HDPE materials, but it is also used with the reinforced thermoplastics. Single-hot-wedge and dual-hot-wedge systems are both available. The dual-hot-wedge weld forms a continuous air channel between two welds. This air channel is used as a means of testing the bond continuity when air pressure is injected into it. Welding rate (movement of the machine) as well as temperature and roller pressure are adjustable and continuously monitored. Adjustments are made according to environmental conditions such as ambient temperature and moisture.

The hot-wedge method has been used in both the factory fabrication of panels and in field installation. It is particularly suited to long, continuous, straight seams. However, without special modification, it does not appear to be suitable for making repairs because of the irregularity of the shapes required to patch liners.

Heat Sealing--

In this thermal seaming method, the heat required to melt and bond the two layers of thermoplastic is applied through the sheets by clamping them between a pair of jaws which are quickly and reproducibly heated, normally by passage of an electrical current through a resistance wire. The sheets remain clamped for a preset period following cessation of the current and the molten polymer solidifies to form an lap bond (Bikales, 1971, p 158).

The advantage of the heat-sealing method is that the complete bonding cycle is readily controlled by a timer and, thus, seams can be made rapidly and reproducibly. As exposure of the heated plastic to air is minimal, the problem of oxidation and embrittlement is reduced.

Another form of heat sealer not sharing the advantages of the clamp type is a heated roller which can be used manually to simultaneously press and melt together both sides of the seam (Skeist, 1965, p 482). Both roller and clamp heat sealers share a serious disadvantage in that heat must pass through the seam and, thus, are generally limited in application to relatively thin sheetings. With thicker sheetings, the bonding process is very slow and the heated surfaces tend to become fluid, flow, and thin down before the bonding surfaces are sufficiently molten for fusion to occur.

Dielectric Seaming--

In dielectric seaming, heat is generated internally within the pieces of sheeting to be joined by directing electromagnetic energy in the radio-frequency region to the seam. The energy field oscillates and causes the permanent or induced dipoles in the polymer to oscillate with the same frequency, creating internal friction and heat. Advantages of dielectric

heating are that the entire cross section of the sheeting is heated quickly and uniformly, the heating process can be instantly started and stopped, the method is very efficient as it does not generate waste heat, and the process is readily controlled and highly reproducible. Pressure is applied until the applied until the area being seamed has cooled and a strong bond formed.

Dielectric seaming can only be used with FMLs based upon thermoplastic polymers synthesized from easily polarizable monomers. The presence of water in an exposed FML can result in internal blowing and sponging of the FML. This technique is suitable only for factory operations where the environmental requirements of the equipment can be met and cannot be used in the field. FMLs that can be seamed by this technique are based on such polymers as polyvinyl chloride, chlorinated polyethylene, and chlorosulfonated polyethylene; polyethylenes cannot be seamed by this technique. Within these limitations, dielectric seaming provides very rapid and reliable seaming (Bikales, 1971, p 161), but it is not suitable for field repair of FMLs.

Solvent Methods

Because of the solubility of noncrystalline thermoplastic polymer compositions in appropriate solvents and the lack of crosslinks, an FML based on a noncrystalline thermoplastic polymer can be seamed with solvent mixtures or with solvents in which the liner compound has been dissolved to form a "bodied solvent." Seaming by these techniques is described below.

Solvent Welding--

Solvent "welding" of noncrystalline thermoplastic sheetings can be achieved by coating the mating surfaces of the sheetings with a solvent or a mixture of solvents suitable for the compound. The two surfaces are then pressed together firmly, e.g., by "stitching" with rollers on a firm base. The time for such a seam to "cure" or set up ranges from 5 minutes to an hour, depending on the type of sheeting and environmental conditions. A few days are usually needed for the solvent to evaporate completely from the seam and for it to achieve full strength. Though this method can be used both in the field and in the factory, it is sensitive to weather conditions, e.g., temperature, humidity, and wind. Volatile solvents which may be desirable at lower temperatures will evaporate too quickly at higher temperatures or may fail under humid conditions to yield an adequate bond because of moisture condensation (Haxo, 1983, p 97).

In forming seams by the solvent-welding method a solvent or blend of solvents must be chosen for the specific plastic to be bonded (Bikales, 1977, p 125; Skeist, 1965, p 434). The solvent must soften and impart tack to the sheeting but not totally dissolve it. The choice of an appropriate solvent is facilitated by knowledge of the volatility and solubility parameters of the solvent and of the solubility parameters of the liner composition (Barton, 1975). In making repairs, it is also necessary to change or

refresh the exposed surface to remove dirt, exudation from the sheeting, e.g., waxes, and moisture. The surface may need to be abraded and buffed to remove an oxidized layer which may not be soluble.

Bodied Solvents--

The use of a bodied solvent to seam thermoplastic sheets is an adaptation of the solvent "welding" method described above. A bodied solvent is a solution of the liner compound to be seamed in a mixture of solvents. The "adhesive" is applied to both surfaces and the two surfaces are pressed together after becoming "tacky." There should be no surface "skinning" or drying of the adhesive when the two surfaces are joined.

The major advantage of a bodied solvent over a straight solvent is the increased viscosity of the solution which allows more control of the evaporation of the adhesive and aids in making seams on a slope. Another advantage of bodied solvents is that the dissolved polymer fills voids or imperfections in the surface of the sheeting and thus improves the consistency and strength of the seams. As with solvent "welding," bodied solvents can only be used with thermoplastic materials that can be dissolved in a suitable mixture of solvents.

The bodied-solvent technique can be used to seam sheetings in the factory and is particularly useful in the field (Haxo, 1983, p 97; Bikales, 1971, p 132). It has been used considerably in the seaming of CSPE, CPE, and PVC membranes and in making field repairs during the installation of these membranes. Testing of seams must wait until the solvent in the seam has evaporated through the membrane or has been driven out by heat.

Adhesives

Adhesives are compounded to have special properties and have been used to seam various types of FMLs. For instance, adhesives that crosslink with time are required to seam vulcanized rubber sheetings. Other adhesives impart "tack" which is useful in seaming. Basically, adhesives depend on a chemical bond with the sheeting to achieve adhesion. Two types of adhesives used in seaming new sheeting and which may be useful in repairs are described below.

Vulcanizing Adhesives--

Vulcanizing adhesives, which are required for seaming vulcanized sheeting, achieve their strength from the crosslinking of the polymeric base. Crosslinking of the polymer in the adhesive may be either a long- or short-term process and may occur under service conditions. Usually, a vulcanizing adhesive is composed of a two-part system, one part containing the polymeric base, and the other part the crosslinking agents. The two components are mixed in the proper ratio before application to the FML. A complete system, as supplied by the manufacturer, usually includes a two-part cement, a rubber-base gum tape, and a lap sealant. Generally, the

degree of crosslinking of the adhesive after cure is significantly lower than that of the sheeting itself; consequently, the adhesive layer is more sensitive to waste liquids and may have to be tested in the specific liquid.

Formulations are generally made specifically for a given application, but some general purpose systems based on butyl rubber (Haxo, 1983, p 99; Skeist, 1965, pp 221-228), neoprene, polysulfides (ibid., pp 286-297), and epoxies (ibid., pp 323-332) have been used. Surface preparation is of major importance, particularly in repairs, in order to achieve a good chemical bond between the adhesive and the membrane substrate.

Contact Adhesives--

Contact adhesives differ significantly from bodied solvents in that the solvent component is evaporated before, rather than after, seam formation (Skeist, 1965, pp 229-247). Solvent volatility is thus a less critical consideration. Contact adhesives are applied to surfaces of sheetings that are to be bonded and then allowed to dry to a "tacky" and solvent-free state before the two surfaces are joined. The use of this type of adhesive requires careful alignment of the lining material before bonding because the joined surfaces should not be realigned after assembly. After joining, the seam should be rolled with a steel or plastic roller in a direction perpendicular to the edge of the seam.

Seaming methods using contact cements share an advantage with vulcanizing adhesives and tape adhesives over the heat and solvent based methods in that they are suitable for use with highly crosslinked sheetings. A disadvantage of contact cement is the "tacky" nature of the surfaces prior to seaming makes them excellent collectors of dust and grit. This factor and the seam alignment problem make contact adhesives more suitable for use in a controlled factory environment than in the field (Haxo, 1983, p 99).

Tape Adhesives

Pressure-sensitive adhesive tapes were used in the past to seam membrane lining materials, particularly low-density polyethylene film, in the field. These tapes are made with pressure-sensitive adhesive applied either to both sides of a flexible substrate or to a flexible backing. In the latter case, the flexible backing is removed once the tape has been placed on one of the surfaces to be joined. More recently, adhesive tapes have been used to position the sheetings while another seaming technique is used to make the permanent bond. As tape adhesives are sensitive to many liquids, the use of tapes alone for permanent seaming or repairing liners for waste disposal facilities is not recommended (Haxo, 1983, p 100; Skeist, 1965, pp 584-592). However, these tapes can be useful in repairing a liner to aid in positioning the sheeting for a permanent bond, e.g., a fillet extrusion seam.

TESTING THE QUALITY OF SEAMS

Critical to the functioning and durability of a polymeric FML in service is the integrity of the seams between assembled sections of the sheeting. The same criticality holds for repairs that are placed on seaming of FMLs during installation. The testing of the seam during the installation of the liner and the associated quality assurance/quality control that is followed are essential to assure the ultimate quality and integrity of the liner system. A repair which involves the seaming of patches should be subjected to the same type of testing and inspection. Unfortunately, the use of "plugs" to make a repair cannot be subjected to these tests. Testing of plugs can only be performed on the final repair over a long period of time or possibly through the use of an hydrostatic test method.

Various tests used to assure the liquid tightness and long-term durability of seams made during liner installation are presented in Table 5. These tests and their applicability to testing repair seams are discussed briefly in the following paragraphs.

TABLE 5. TESTS TO DETERMINE THE QUALITY OF FML SEAMS

Chemical compatibility of seams with waste liquid
Nondestructive testing of actual seams
- Visual inspection
- Mechanical probes
- Air lance
- Vacuum box
- Ultrasonic methods
- Pressurization of space between dual seams
- Electrical sparking
- Testing of coupons made alongside actual seaming
Destructive testing of samples cut from liner seams
- Shear tests
- Peel tests
- Dead load tests

The first type of seam test is for determining the chemical compatibility of the seams by immersion in the waste medium and measuring the effect upon adhesion. These tests should be performed before the actual seaming, either in the plant or the field. In the case of a repair, such tests should be run on sample seams of the exposed liner in order to select an adequate seaming procedure.

The second type includes nondestructive tests that are performed on the liner seams that have been placed in the field. In this testing the actual seams are not cut, but are tested without disturbing them. One of the test procedures involves preparing coupons during the actual seaming operation, using the same equipment and seaming procedures used in making the actual seams.

The third type of test is destructive testing which is performed on samples of the seam cut from the actual liner. This type of testing requires the placement of patches over the holes from which the samples have been cut.

Chemical Compatibility of Liner Seams with Waste Liquid

The long-term durability of a seaming method should be determined before it is used to seam a liner. To measure durability, samples of typical seams are immersed for extended periods of time in the waste liquid to be confined by the liner. After each exposure period, the samples are removed and tested. Before repairing a liner, durability determinations of seams should be made using samples of the exposed sheeting because the lining material that is to be repaired may have absorbed components of the waste liquid to which it has been exposed. These absorbed components can make adequate adhesion difficult or even impossible to accomplish without resulting in loss of integrity or inadequate performance of the liner.

Nondestructive Test Methods

There are several available methods for qualitatively testing seams and repairs without cutting samples from a continuous liner. Some of these nondestructive test methods can be used to measure the continuity of a seam but cannot be used to measure the relative strength of the seam or its durability in a waste liquid environment. Nondestructive methods should be used in conjunction with destructive testing in a quality assurance program.

Visual Inspection--

All seams fabricated in the factory and the field should be visually inspected. Similarly, such an inspection should be made on all repairs.

Air-Lance--

In the air-lance method, a jet of air is applied to the upper edge of an overlap seam in an effort to detect a nonbonded area. When correctly used, the impinging air stream will inflate a nonbonded area. This area is then marked by the inspector for subsequent repair.

Mechanical Probe--

To detect nonbonded areas, an inspector can slide a dull tool, such as a blunt screwdriver, along the top edge of a seam. A nonbonded area can be

easily lifted and then marked for repair. Care must be exercised when using this method so as not to puncture the FML or use excessive force against the seam edge. This method (sometimes called the "pick test") will detect edge nonbonds that the air-lance method may not detect because of a lightly tacked down edge. Coupled with visual inspection, this method can be a rapid test for seam and repair continuity.

Vacuum Box--

The vacuum box test for seam integrity consists of applying a partial vacuum above a section of the seam which has been covered with a soapy solution. A nonbonded area is indicated by bubbles when the vacuum is applied. The equipment used in this test consists of a vacuum box which can be sealed to the seam area and which has a glass viewport. The vacuum is supplied by a portable vacuum pump.

It would appear that the vacuum box method can be used in testing some repairs if a seal to the repaired liner can be made.

Ultrasonic Devices--

Ultrasonic methods have been found useful in detecting nonbonded areas in seams of FMLs, particularly in testing seams of HDPE liners. The Ultrasonic Pulse Echo Technique is presently used in the field for testing continuity of HDPE flat-fusion welds. However, this method works equally well on other unreinforced FMLs. To assure good bonding across the seam width, the entire seam surface area must be scanned with the ultrasonic pulse echo transducer. The operator visually marks all nonbonded areas for repair or capping. This method, although not yet widely used, can be applied to reinforced as well as unreinforced FMLs as the scrim reinforcement does not seem to affect the low frequency transmission.

Pressurized Dual Seam--

The pressurized dual seam method is used on a dual-wedge thermally-welded seam where an air channel exists between two bonded lengths of seam. After the dual-wedge seam has been fabricated for a given length, both ends are sealed. A needle with attached pressure gage and air valve is inserted into the air space between the thermally-welded areas, and pressure is applied to the air channel. The gage is monitored for drop in air pressure over time as an indicator of a seam nonbond and leak. Although this method tests long lengths of seam at one time, if a leak is detected, that entire length must be rechecked with a vacuum box or the length must be cap stripped. The pressurizable dual seam is presently in use on thermally-welded HDPE but could be used on other FMLs capable of being seamed with a dual-wedge welder.

Electrical Spark--

Electrical spark methods have been effectively used to detect voids and pinholes or nonbonds, primarily in the semicrystalline thermoplastic

welds. Basically, a high voltage (15-30 kV) electrical current is applied to a seam area and any leakage to ground can be detected by sparking. A ground wire can be designed into the FML system and attached to the back of a seam for subsequent use of the spark test apparatus.

Testing of Seam Samples Made Alongside Actual Seams--

The preparation of seam samples alongside the seam during the actual seaming operation, using the same procedure, materials, and equipment, has been used as a means of assessing the quality of seams. The samples that are thus made are then tested for peel and shear similarly to that described below in the section on destructive testing.

Testing of seam samples made alongside actual seams appears to be suitable for testing repairs made in essentially dry and accessible areas; however, the samples prepared for testing must be of essentially the same composition as the FML substrate being repaired. Because of changes in the composition of the FML with time, this requirement will result in taking a sample out of the in-place liner, thus enlarging the hole or other damage to be repaired. Such a test would appear to be almost impossible to perform in repairing damage below the waste.

Destructive Test Methods

In addition to nondestructive test methods that test for seam continuity or repair integrity, destructive test methods that involve testing samples cut from the in-place seams are also needed to determine their actual strength. Seam testing can be performed in two modes, i.e., in peel and in shear. In order to obtain representative seam data on a sample of a particular seam, it is important that peel specimens and shear specimens are died out alternately along the length of the seam sample. The sample often used for destructive testing ranges from 18 to 24 inches of seam length, which poses a problem in the destructive testing of repairs because the hole resulting from the sample removal may be larger than the repair area.

Shear Strength Testing--

Shear strength testing is performed by applying a force across the seam in a direction parallel to the plane of the bond, thus subjecting the bond interface to a shearing force. In most specification testing, a constant rate of extension testing machine is used; however, in some on-site testing during installation, manually powered screw-type devices have been used.

At present there is no test method intended specifically for testing FML seams in shear. One of the methods most frequently cited for the shear testing of seams made from unreinforced sheeting is ASTM D882, which is a strip tensile test method intended for determining the properties of plastic sheeting less than 1.0 mm (0.04 in.) in thickness. Also cited are ASTM D3083 and D638, either by themselves or in conjunction with ASTM D882. ASTM

D3083 is a specification for PVC sheeting which specifies the use of ASTM D882 for seam testing with some modifications. ASTM D638 is a dumbbell tensile test method intended for determining the properties of plastic sheeting greater than 1.0 mm (0.04 in.) in thickness. All of these test methods need to be modified to be used for shear testing. None of these methods set criteria for the acceptance or rejection of a seam; such criteria need to be set in separate specifications.

The type of specimens that have been used for shear testing of seams fabricated from unreinforced sheeting have included strips 12.5-25 mm (0.5-1.0 in.) in width, D638 Type I dumbbells [which feature a 12.5 mm (0.5 in.) narrow width test area], and D638 Type IV dumbbells [which feature a 6.25 mm (0.25 in.) narrow width test area.] The dumbbell test specimens have been used in cases where it was necessary to localize the tensile stress in the seam part of the sample away from the grips, as in the case of seams fabricated from semicrystalline FMLs. Results are recorded as newtons per meter (pounds force per inch) of seam width at maximum and sometimes also failure, if maximum occurs before failure. The mode of failure (e.g., failure at seam edge, failure at grip edge, failure in seam interface) should be recorded as well as test parameters such as grip separation, crosshead speed, seam width, temperature, and humidity.

Testing of seams made with reinforced sheeting is usually performed in accordance with a modified version of ASTM D751 Grab Method (NSF, 1983). In the modification, the distance between the clamps at the start of test is 153 mm (6 in.) plus the seam width. Total length of the test specimen is 255 mm (9 in.) plus the seam width. Results are reported in newtons (pounds force).

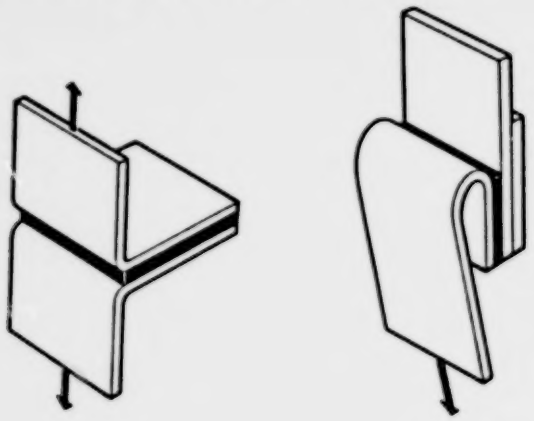
Peel Testing--

Peel testing is performed by applying a load such that the bonded interface is subjected to a peeling force that attempts to separate the two FML sheets that have been seamed together. Peel testing of all types of FMLs is usually performed in accordance with ASTM D413 at a separation rate of 5 mm/ min. (2 ipm). Testing can be performed either in 90° or 180° peel (Figure 1). Peel testing of semicrystalline FMLs in 180° peel is difficult to perform because of the stiffness of these materials.

On testing seams fabricated from reinforced FMLs, 25 mm- (1 in.-) wide strip specimens are usually used. On testing seams fabricated from semi-crystalline FMLs, D638 Type I and Type IV dumbbell specimens have sometimes been used to localize the peeling force in the seam test area.

Test results can be either a maximum or an average peel value. ASTM D413 requires the average value over the seam test area, but in cases in which the seam test specimens break through one of the FML sheets or through a weld bead rather than delaminate along the contact interface between the two sheets, often only a maximum value can be reported. Results are reported in newtons per meter (pounds force per inch) of seam width. The type of

value (i.e., maximum or average), as well as the mode of failure and other applicable test parameters, should also be reported.



(a) 90° peel (b) 180° peel

Figure 1. Two configurations of peel testing.

Peel testing using a static or "dead load" at room temperature, that is, 23°C (73.4°F), and at elevated temperatures [usually 50°C (122°F) or 70°C (158°F)] can provide a good indicator of time-dependent weaknesses that will not be observed under dynamic testing. Dead load testing at elevated temperatures can be used as a method of revealing the sensitivity of a seam system to long-term exposures on the FMLs and the seaming system.

SECTION 5

LITERATURE SEARCH AND CONSULTANTS MEETING ON LINER REPAIR AND ADHESION

INTRODUCTION

This section presents the methodology and the results of the searches of the open technical literature for information on seaming and repairing FMLs. The information available in the open literature on adhesion and adhesives as it relates to repairing FMLs is summarized. Also presented is the information that was made available and discussed at a meeting of the consultants to the original project.

LITERATURE SEARCH ON LINER REPAIR AND ADHESION

In the original project, the open technical literature through 1982 was searched for information on methods of seaming and repairing FMLs in general use in waste impoundments. The literature was searched both by hand and by computer. The data bases and libraries included in this search were:

- Matrecon's library.
- Four data bases on the Lockheed Dialog system.
- The Applied Science and Technology Index.

A total of 211 citations to seaming and to FMLs for waste disposal facilities were found, of which 17 were used in the bibliography.

Matrecon's card catalog was searched by hand using the subject categories: adhesion and adhesives; adhesion and bonding; linings; canal linings; membranes. Fifteen citations were found and 14 were used.

Four computer data bases were searched on the Lockheed Dialog system: NTIS (1964-83); COMPENDEX (1970-82); ENVIROLINE (1971-82); and CA Search (1982). The search strategy combined variations of "plastic" (including "polymer" and "rubber") with variations of "repair" (including "bond", "adhesive", "weld", and "seal") with "waste". The method of accessing the data bases was such that all variations of the root word were pulled out. For example, the request for citations on "adhesive" also pulled out citations for "adhesion". Using this search strategy, a total of 190 citations were found and evaluated. Of these only three were actually used in the bibliography.

The Applied Science and Technology Index (1972-82) was searched by hand at the University of California using the subject categories: plastic sheeting; rubber lining; plastic lining; plastic films; polymer; bond; repair; weld; adhesion; and seal. Six citations were found, but none were new or pertinent.

Overall, even though the literature on adhesives and adhesion of polymeric materials is voluminous, the references in the open technical literature that bear directly on the repair of in-service FMLs are almost nonexistent. Also, even though each FML supplier and installer has developed methods of repairing damaged or defective liners during installation, they have not developed methods specifically for repairing in-service FMLs except for weather-exposed surfaces above the waterline. Thus, little information was available from the liner industry.

ADHESION OF FMLS

In considering permanent repairs to damaged, defective, or aged FMLs, it would be necessary to apply to the repair the same criteria and testing that are applied to a liner that is being installed. Examples of such criteria for factory seams made in preparing panels of an FML are set forth in National Sanitation Foundation Standard Number 54 (NSF, 1985). Similar criteria are also set forth for field seams in the EPA Technical Resource Document SW-870 (Matrecon, 1983), in EPA guidance documents, and in project specifications written by designers of waste disposal facilities. Basically, the criteria for seams in an FML are a film-tearing bond when the seam is made (i.e., the seam will break in the parent membrane material and not at the interface between the two seamed sheets when tested in shear) and a film-tearing bond after exposure to weather or to the waste liquid. For thermoplastic FMLs, this means that the bond should not fail in an adhesive mode when tested and that the interface between the two layers being joined is eliminated to yield a monolithic structure. It is recognized that mechanical seals, in which the layers are pressed together and pressure is maintained, may also be leakproof without adhesion between the layers. Except for the use of plugging materials, mechanical seals were not considered in this project. This section summarizes the information in the open technical literature on the requirements for a good adhesive bond and then discusses these requirements with respect to the service conditions of in-place FML.

In reviewing the references from the voluminous literature on adhesion (for example, Bikales, 1971; Houwink and Salomon, 1965 and 1967; Mark et al, 1964-77; Skeist 1965; Snogren, 1974), it was quite apparent that it would be difficult if not impossible to meet the conditions required to make sound bonds between either a patch or a plug and an in-service FML, except possibly on those portions of an uncovered liner on a slope exposed only to the weather. These references reiterate the need for proper preparation of the surfaces to be bonded, including the need for cleanliness and dryness, in achieving good adhesive bonding. In addition, different types of bonding

may require different types of surface treatment. For instance, proper surface preparation before the use of an adhesive may differ from proper surface preparation before the use of either a thermal or solvent-based welding technique.

Thelen (1962) lists a series of objectives for preparing the substrate surfaces prior to achieving a good bond, whether they be patches or plugs. Those objectives that pertain to FMLs are listed in an adapted form below. Surface preparation of FMLs is performed for one or more of the following reasons:

1. To remove dust, scale, soils, and liquids that could trap air or prevent either an adhesive from contacting a "solid" surface or the surfaces of the two FML sheets to be welded together from contacting each other. Waxes and other chemicals that "bloom" to the surface of an FML should also be removed as these species can result in a monomolecular layer that is not chemically active and will prevent bonding with another surface.

2. To etch or roughen the surface thereby giving an adhesive more surface area and more interlock with the substrate.

3. To plane, grind, or otherwise smooth rough or bumpy surfaces so that they can be brought into closer contact with one another and, if an adhesive is being used, so that an adhesive will flow better on them.

4. In the case of chemically inert materials, such as vulcanized FMLs, to treat the surfaces chemically, for instance, to oxidize the surfaces so as to create chemical moieties that are reactive with adhesives. Such surface treatment of polyethylene can be performed under factory conditions to bond polyethylenes to other materials with adhesives.

5. To dry the surfaces, not only to remove gross layers of water but to remove as much as possible of any adsorbed films.

6. To prime the surface after it has been been prepared for bonding so that the substrates can be stored without fear of recontamination before they are bonded.

7. Following any chemical treatments for preparing the surface for bonding, for example, chemical etching, to rinse the surfaces thoroughly to remove residual chemicals or soluble salts that would inhibit bonding.

The different reasons for preparing a surface all indicate the need for control over the conditions under which the seaming or repairing operation takes place. In a factory seaming operation, conditions are usually optimized. Environmental factors such as temperature extremes, wind, humidity,

rain, dust, and sunlight can be significant in a field seaming operation in the manner in which they affect the procedures for surface preparation and for seaming. One result is that some seaming methods suitable in a factory environment are eliminated from use in the field.

Given that seaming during installation is being performed under environmental conditions that do not significantly affect the seaming procedure, the most important aspect of seaming will involve keeping the sheeting free of surface contamination from dust and grime. This contamination may be removed with a brush or cloth or, in the case of heavier gauge materials, ground away with a hand electric grinder (Frobel, 1984).

In addition to proper surface preparation, there are other important requirements for making a successful seam or repair in an FML. In repairing a thermoplastic FML using either solvent- or thermal-welding techniques, it is important that the patch and the FML essentially have the same composition. Differences in the solubility parameters of the polymers, the plasticizer contents, and the amount of crystallinity can result in inadequate bonds. For example, a polypropylene sheeting cannot be welded with an HDPE sheeting, and various types of polyethylenes cannot be welded to each other. The choice of a particular bonding system or procedure requires compatibility with the chemical properties of the materials to be bonded. Exposing liner materials to the environment and to wastes results in changes in the liner which are often not limited to the surface. These changes in the properties of a liner may prevent successful use of the procedure used to seam the liner when it was being installed. The effects of exposing FMLs to waste liquids are discussed in more detail in Section 6.

CONSULTANTS' MEETING

Early in the original project, a consultant group was formed and a meeting on the project was held at which the general subject of repairability of FMLs was discussed. Particular emphasis was given to repairing in-service FMLs that had been damaged below the waste surface or below the waterline.

Reservations regarding the viability of liner patching as an *in situ* repair procedure were reinforced by the consensus of attendees. They agreed that bonding patches to liners exposed to wastes was generally not feasible due to the tendency of most liner materials to absorb water and solvents and due to the uncertainties and difficulties involved in removing these contaminants. As a result, it was recommended that investigation of patching in this project be limited to the repair of above-surface liner damage and defects in HDPE FMLs. Laboratory and field data have shown that HDPE materials have relatively little tendency to absorb solvents and wastes.

For other lining materials and, in the case of subsurface repairs, plugging methods were felt to be a much more suitable approach for repairing

in-service FMLs. Some systems, notably epoxies, have been commercially formulated specifically for sealing and plugging in wet or underwater environments. These systems have been used to protect structures exposed to corrosive environments and to plug leaks in caissons and reservoirs, even in an environment of water flowing under pressure.

In the case of a waste impoundment liner repair, these materials could stop the flow through a defect by physically plugging it with a volume of relatively high viscosity liquid, which would subsequently cure and harden. Good adhesion to the liner surface would be desirable but is not essential in plugging a defect if the defect is embedded in the plug and if there is a good mechanical seal.

REPAIR OF FMLS IN WASTE DISPOSAL SITUATIONS

During the installation of an FML liner system, it is routinely necessary to repair the liner by patching. For example, when a sample is cut from the liner to test seam quality, the resultant hole must be repaired. Generally, the same method used to make the original seams is used to make these repairs.

Several examples of repairing in-service liners are briefly discussed below. These examples indicate the difficulties and limitations of available repairing methods.

1. One consultant at the consultants' meeting described the successful patching of a CSPE liner and a CSPE floating cover. Both repairs were made on materials that had been exposed to the weather but not to waste or water. The damage to the liner was on the berm. Care was taken to prepare the liner surface by thorough cleaning with trichloroethylene, and the patch was bonded to the liner with a CSPE adhesive. Repair to the floating cover was performed by bonding a patch to the cover with a two-part neoprene adhesive.

2. An attempt was made to repair a hole, approximately 18 in. x 18 in. in size, cut in a PVC liner (Haxo et al, 1982). The hole was cut to remove a sample to assess the effect of 6 years of exposure to MSW leachate at an EPA demonstration project. In spite of the access to the liner, a satisfactory repair of the hole could not be made using a patch from a sheeting reported to have been a retained sample of the original PVC liner and a solvent-welding system of toluene and tetrahydrofuran. The flow of water in the area could not be stopped. The ground surface on which the liner rested was irregular and became quite soft as the work progressed. The patch material was thicker and stiffer than the original liner and was too stiff to conform to the irregular area of the attempted repair. In addition, the solvent had no tack to hold the patch to the liner and quickly softened the PVC liner so that no adhesion was possible.

3. A sprayed-on asphalt liner from which a small circular sample was removed for testing and analysis was repaired with hydraulic cement. The seal around the plug did not appear to leak during the one year of exposure (Haxo et al, 1982). This repair is described more fully in Section 7.

4. A procedure for repairing sprayed-on asphalt liners used to line some ponds holding acidic wastes was described to the author. The distressed area was made accessible for inspection and possible repair by letting the level of the pond down and dropping a small caisson-like metal box into this area. After draining the contents from inside the box, the asphalt surface was partially dried and cleaned. Melted asphalt was then poured into the box to make a seal.

5. The author observed the repair of a small puncture of an HDPE liner. Silicone caulk was used to form a plug. The adhesion of the caulk to the exposed liner surface appeared to be satisfactory, but the plug was tender and needed protection to avoid damage.

6. The author inspected two "repaired," EPDM-lined surface impoundments, both of which held a wastewater that contained small amounts of organics. Some of the original seams in the liners had opened due to softening of the adhesives. Before repairing the liners, the impoundments were emptied and partially dried. The damaged seams were then covered with strips of EPDM sheeting which were bonded to the liner with the same adhesion system used to make the original seams. At the time the author inspected the impoundments, the strips under the water had completely separated from the liners, but the strips on the slope and berm had maintained good adhesion to the liners.

7. Polyurethane foams have been investigated by Mitchell et al (1973) for plugging damaged metal drums that contained wastes and other hazardous liquids. This procedure, which showed promise, is described in Section 7.

SECTION 6

REPAIR OF DAMAGED EMPLACED FMLS

REPAIR OF FMLS DURING DIFFERENT PHASES IN THEIR SERVICE LIFE

The need for repairing FMLs may occur at any time during three major phases in the service life of the liner:

- During and immediately after installation. Repairs at this time should include repair of damage that took place during installation, patching holes that are required for field sampling and inspection, and repairing defective seams which show up during inspection and quality assurance testing.

- Shortly after installation when the liner may have been partially covered with soil or undergoing performance-type inspection.

- After the facility has been placed in service and the liner has been in contact with wastes and covered with soil. This period ranges from a few months through the rest of the service life of the liner.

During the first phase, at the time of installation, the FML is generally only a few weeks old and has aged little. Efforts are made to ship the material from the factory to the field in as fresh a condition as possible. Many FMLs are shipped in cartons that have been wrapped in polyethylene film to minimize the absorption of moisture, which may affect the bonding characteristics of some lining materials, e.g., CSPE FMLs. Each FML has its own procedure and materials for seaming as well as methods for field repair of holes and seams. For example, panels of CSPE FMLs are fabricated in the factory, generally by heat-sealing methods, though some solvent methods are still used. In the field, bodied solvents and solvent cements are used to assemble the panels into the liner. Special cleaning of the surfaces prior to seaming the panels is necessary. Repairs are performed using the same bodied solvents or solvent cements used to seam the panels.

In the case of HDPE FMLs, which are of major importance for hazardous waste containment, most of the seaming is done in the field, except in cases where narrow HDPE sheets are produced and assembled into panels or wider sheets in the factory. Various methods of seaming HDPE liners are described in Section 4. In general, the same methods used to seam HDPE FMLs in the field can be used to repair them. For most seaming systems, the surfaces of

FMLs must be cleaned. For example, HDPE surfaces can be oxidized or covered with a "bloom" of constituents in the HDPE that are insoluble and can form a nonadhesive surface. In many situations the surfaces must be buffed and seamed shortly afterward. The same surface preparation performed before seaming should also be performed before repairs are made.

A vulcanized FML, such as one based on vulcanized ethylene propylene rubber (EPDM), is seamed in the factory with vulcanizing equipment. In the field, low-temperature vulcanizing adhesives are used. This type of FML is repaired with the use of adhesives and various vulcanizable tapes placed either between the sheeting or on the edge of the top sheeting.

In the second phase, shortly after installation, the FML is somewhat older though not exposed to the weathering to such a degree that aging has become an important factor in performing repairs. However, after limited exposure to the weather and the soil cover, the sheeting may have become dirty. The success of a patch-type repair will depend on achieving the necessary cleanliness and freedom from contamination. In addition, if leaks have been detected during performance-type testing, the causes of these leaks, i.e., holes or inadequate seams, may not be easily located. Consequently, a great deal of effort may be required to locate the defects. Repairs may require large patches using the seaming techniques that are specific to the FML.

The third phase covers the principal service life of an FML which can range from a few weeks to the closure of the site. Many years can pass before leakage is observed. The repair of leakage in surface impoundments generally requires the impoundment to be drained, cleaned, and dried before repairs can be made. Under the conditions of exposure, the liner may have changed considerably in composition and mechanical properties. Successful patching of a liner requires the surfaces to be clean and free of moisture and other contamination, a condition which is sometimes difficult, if not impossible to meet in the field. In the areas of an impoundment which are normally exposed only to the weather, repairs in many cases can be made without draining the impoundment and generally the repair is effective. Sections of FMLs that are in service below the waterline may have swelled or stiffened. For instance, plasticizers may have been extracted. Thus, the compositions may be uncertain. The use of heat guns may not be possible because of the volatility of some of the organic constituents in the waste liquid which may have been absorbed by the FML. Also, there must be a close matching on a molecular scale of the compositions of the patching material and of the damaged FML that is to be repaired. It should be pointed out that, even though the repairability of an FML may be greatly reduced by the absorption of small amounts of water, oil, and organics, the overall integrity or quality of the FML sheeting itself probably remains essentially unchanged.

Table 6 summarizes the different types of defects or damage that may occur and the different types of repair or remedy that are available during these three phases.

TABLE 6. DAMAGE AND POSSIBLE REPAIR OF FMLS IN DIFFERENT PHASES OF SERVICE LIFE IN DIFFERENT TYPES OF DISPOSAL FACILITIES

FML type and polymer	Phase of service life: Construction/installation to acceptance — Seaming methods	Construction/installation to acceptance — Types of defect or damage	Construction/installation to acceptance — Types of remedy or repair	Short-term service — Types of damage	Short-term service — Types of remedy or repair	Long-term exposure to waste — Types of damage	Long-term exposure to waste — Types of remedy or repair
Crosslinked polymers[a]							
(e.g., ethylene propylene rubber, butyl rubber, neoprene, epichlorohydrin rubber, chlorinated polyethylene, and chlorosulfonated polyethylene)	Vulcanizers (factory made seams) Crosslinkable adhesives	Punctures Tears Cuts Delamination or ply separation Seam openings or "holidays" Samples cut for seam tests	Patch Cap strip (over seam) Remove and replace damaged sections Remove and replace liner	Open seams Delamination or ply separation Tears and splits Punctures Pinholes	Clean and dry, then patch or plug liner or cap seams	Delamination or ply separation Tears Punctures Opening of seams Severe swelling Creep and holes Ice damage Dike damage	Surface impoundment: - If above waterline, drop level, clean, then patch or plug - If below waterline, drop level, clean out, water wash, dry out, patch or plug after buffing and solvent wash - Close unit Landfill: - Close unit Waste pile: - Cleanup - Close unit
Thermoplastics[a]							
(e.g., polyvinyl chloride, polyvinyl chloride--oil-resistant, chlorinated polyethylene, and chlorosulfonated polyethylene)	Thermal methods: - Heat gun - Hot wedge Solvent methods: - Solvents - Bodied solvents - Solvent cements Adhesives Mechanical	Punctures Tears Cuts Delamination or ply separation Seam openings or "holidays" Samples cut for seam tests	Patch Cap strip (over seam) Remove and replace damaged sections Remove and replace liner	Open seams Delamination or ply separation Tears and splits Punctures Pinholes	Clean and dry, then patch or plug liner or cap seams	Delamination or ply separation Tears Punctures Opening of seams Severe swelling Creep and holes Ice damage Algae damage (surface impoundment) Dike damage Damage from dissolution Stiffening and cracking Massive tears resulting from slippage of soil cover	Surface impoundment: - If above waterline, drop level, clean, then patch or plug - If below waterline, drop level, clean out, water wash, dry out, patch or plug after buffing and solvent wash - Close unit Landfill: - Close unit Waste pile: - Cleanup - Close unit

Continued. . .

TABLE 6. CONTINUED

FML type and polymer	Phase of service life: Construction/installation to acceptance — Seaming methods	Construction/installation to acceptance — Types of defect or damage	Construction/installation to acceptance — Types of remedy or repair	Short-term service — Types of damage	Short-term service — Types of remedy or repair	Long-term exposure to waste — Types of damage	Long-term exposure to waste — Types of remedy or repair
Semicrystalline thermoplastics							
(e.g., polyethylenes[b], and polyester elastomers[a])	Thermal methods: - Flat extrusion - Fusion-welding - Fillet extrusion - Wedge - Heat gun	Punctures Tears Cuts Delamination Seam openings or "holidays" Samples cut for seam tests Cracks	Patch Cap strip (over seam) Plug of molten resin Remove and replace damaged sections Remove and replace liner Cover with second liner	Open seams Delamination Tears, splits Punctures Pinholes Environmental stress cracks Fatigue cracks	Clean and dry, then patch or plug liner or cap seams	Delamination Tears Punctures Opening of seams Creep and holes Ice damage Dike damage Environmental stress cracks Fatigue cracks	Surface impoundment: - If above waterline, drop level, clean, then patch or plug - If below waterline, drop level, clean out, water wash, dry out, patch or plug after buffing and solvent wash - Close unit Landfill: - Close unit Waste pile: - Cleanup - Close unit

[a]Unreinforced and fabric-reinforced FMLs.

[b]Unreinforced only.

EFFECTS OF EXTENDED EXPOSURE OF FMLS ON THEIR REPAIRABILITY

Exposing FMLs to the environment and to wastes for extended periods can result in changes in the liner that are not limited to the surface, but still can affect the ability to achieve adhesion between an FML and a patch or a plug. Exposure to the environment alone can cause significant chemical and physical changes. Plasticizers and additives may decompose or volatilize (i.e., evaporate) with time and exposure to heat, oxygen, ultraviolet radiation, or biochemical breakdown. The base polymer itself is also affected by exposure to heat, oxygen, and ultraviolet radiation. Prolonged exposure may lead to the gradual loss of liner physical properties through swelling, embrittlement, or even disintegration.

Exposure to wastes or water alone is also known to alter liner physical properties. Tables 7 and 8 present data on the absorption of water, of municipal solid waste leachate, and of eight industrial wastes by a variety of commonly used FMLs. After one year of exposure, water absorption ranged from 0.2% increase in weight for a polyethylene liner to 18% increase in weight for a chlorosulfonated polyethylene material. Exposure to the oily "Weed Oil" waste for 809 days led to a 370% weight gain for a chlorosulfonated polyethylene and the apparent dissolution of a chlorinated polyethylene.

Swellings of this magnitude indicate that significant interactions have occurred between the liner and waste and that the chemical composition of the FML has actually changed. Components of the waste have swollen, diluted, and weakened the liner and perhaps even extracted some of its components. The effects of a sequence of waste exposures or of combined waste and environmental exposures would be expected to lead to even greater changes in the liner.

It is clear that attempts to repair an in-service liner *in situ* by seaming a patch to a liner are subject to at least two complications: (1) the liner surface is virtually certain to be heavily contaminated through waste exposure and (2) actual changes may have occurred in the liner composition through its entire cross section. These complications would be specific to each individual repair location and would make general recommendations as to proper seaming procedures unreliable. For instance, in the case of patching procedures for thermoplastic liners using thermal methods (e.g., heat gun, welding, fusion), the water universally present in waste impoundments would prevent bonding. The rapid application of heat would cause the water to explode into steam, disrupting the bond and possibly damaging a section of the liner.

As indicated above, only a small amount of absorbed liquid, whether water or an organic, can seriously reduce the repairability of a liner without significantly damaging the FML sheeting itself.

TABLE 7. WATER AND LEACHATE ABSORPTION BY POLYMERIC FMLS[a]

(Data in percent absorbed by weight)

Polymer	Liner number[b]	Immersed in water at room temperature for one year	Immersed in municipal solid waste leachate for one year
Butyl rubber	7	1.60	1.78
	22	1.70	2.32
	24	1.10	1.0
Chlorinated polyethylene	12	13.10	9.0
	13R	19.60	12.4
	23	15.50	10.3
Chlorosulfonated polyethylene	3	17.40	20.0
	4R	18.00	19.0
	6R	9.20	13.6
	14R	11.20	8.71
Ethylene propylene rubber	8	1.40	5.95
	16	4.80	5.50
	25	1.50	5.99
	26	1.60	8.99
Neoprene	9	22.7	8.73
Polybutylene	20	0.25	0.33
Polyethylene (low-density)	21	0.20	0.25
Polypropylene	27	0.28	0.40
Polyvinyl chloride	10	1.85	6.72
	11	1.85	5.0
	15	2.10	4.64
	17	1.85	3.29
	19	0.60	0.75

[a]Matrecon, 1983, p 150.

[b]Matercon identification number; R = fabric reinforced.

TABLE 8. ABSORPTION OF WASTE LIQUID BY POLYMERIC FMLS ON IMMERSION IN SELECTED WASTES

(Data in Percent Change in Weight)

Polymer	Liner number[a]	Data on liner compound		Waste and immersion time in days							
		Type[b]	Extractables, %	Pesticide, 807 d	"HNO_3-HF-HOAc," 751 d	"HFL," 761 d	"Spent Caustic," 780 d	Lead waste blend, 786 d	"Oil Pond 104," 752 d	"Slurry Oil," 761 d	"Weed Oil," 809 d
Butyl rubber	44	VZ	11.8	1.6	3.8	3.7	0.8	28.7	103.9	31.2	64.2
Chlorinated polyethylene	77	TP	9.1	12.7	19.9	12.9	1.1	118.9	36.9	226.4	ND[c]
Chlorosulfonated polyethylene	6R	TP	3.8	17.3	10.0	9.0	4.3	120.7	49.5	105.2	368.4
Chlorosulfonated polyethylene	55	TP	4.1	15.7	10.9	7.7	3.3	116.2	55.0	110.5	347.5
Elasticized polyolefin	36	TP	5.5	0.5	7.6	1.1	0.6	17.0	28.9	29.4	38.1
Ethylene propylene rubber	83R	TP	18.2	4.5	4.2	3.1	1.6	24.8	26.5	19.8	84.4
Ethylene propylene rubber	91	VZ	23.6	10.4	50.9	12.9	1.3	34.7	84.7	34.2	76.2
Neoprene	90	VZ	21.5	11.4	17.4	12.0	1.5	59.1	26.3	142.6	89.3
Polyester elastomer	75	TP	2.7	4.2	6.4	2.0	1.5	7.4	8.5	16.6	14.7
Polyvinyl chloride	11	TP	33.9	5.1	22.1	18.1	0.4	-1.5	-10.4	18.5	15.3
Polyvinyl chloride	59	TP	35.9	1.0	-6.1	0.9	-0.9	7.4	-0.5	28.9	24.7
Polyvinyl chloride	88	TP	33.9	1.6	28.2	14.3	1.1	-5.2	-9.8	14.1	25.2

[a]Matrecon identification number; R = fabric reinforced.

[b]VZ = Vulcanized; TP = thermoplastic.

[c]ND = Specimen was lost; some indication that it dissolved in the waste.

Source: Matrecon, 1983, p 170.

SECTION 7

EXPERIMENTAL WORK

One of the objectives of the original project (EPA Contract 68-03-1714) was to explore experimentally the in situ repair of defects or damage to various FMLs by patching. It was also desired that the repairs be permanent. On reviewing the literature and documentation of current liner and adhesion technologies, it became clear that patching would in many cases not be a realistic repair method. The root of the problem lies in the tendency of FMLs during exposure to waste liquids to absorb even small amounts of water and organic constituents that are in the waste liquids or to lose components of the compound, e.g., plasticizers and antidegradants, to the waste liquids. Consequently, the bonding ability of damaged FMLs to patches and to similar unexposed FMLs may be drastically reduced. This conclusion was supported by the consensus of attendees at the consultants' meeting at the beginning of the original project. It was decided to limit the investigation of patching as a means of repairing a liner to the specific case of the above-surface repair of HDPE liners and to explore plugging methods for repairing lining materials that may have been wet or emplaced in damp environments.

PATCHING OF DAMAGED HDPE MEMBRANES

Surface Preparation Methods

Our experience in the preparation and testing of sample patches on HDPE liners coincided with observations described at the February 1, 1983, consultants meeting and observations made repeatedly in the literature (Skeist, 1965 and Snogren, 1974), that adequate surface preparation is a necessary prerequisite to the proper seaming or bonding of liner patches.

No truly reliable solvent, bodied solvent, dielectric bonding technique, or contact adhesive is available for seaming HDPE sheeting, so testing was limited to seams formed using heat and fusion/welding. The seam quality obtained using either of these methods was comparable and directly reflected the mode of surface preparation used, as discussed in Section 6.

A test program was drawn up to assess the differences in surface preparation of a HDPE membrane. A 40-mil HDPE sheeting (Matrecon liner No. 268) was chosen as a model material and bonded using heat and fusion with a Leister-Kombi Type "Triac" heat gun which might be used for repairing FMLs. Table 9 presents the tensile properties and hardness of this membrane.

TABLE 9. TENSILE PROPERTIES[a] AND HARDNESS[b] OF A 40-MIL HDPE SHEETING[c]

Property	Test value		
	In machine direction	In tranverse direction	Average
Tensile at yield, ppi	101.0	106.0	103.5
Elongation at yield, %	20	15	18
Tensile at break, ppi	171.2	167.5	169.4
Elongation at break, %	795	760	780
Stress at 100% elongation, ppi	77.2	74.4	75.8
Stress at 200% elongation, ppi	77.8	76.6	77.2
Hardness, Durometer points, 5-second reading	...	...	56D
Thickness (average), mil	...	...	37.2

[a]ASTM D638 using a jaw separation rate of 2 inches per minute (ipm) and Type IV specimens.

[b]ASTM D2240.

[c]Matrecon identification No. 268.

The following three surface preparation methods were explored:

- No special surface preparation, except wiping clean of dust.
- Wiping with a solvent (e.g., xylene).
- Abrasion using coarse aluminum oxide paper followed by a solvent wipe.

In all cases, the specimens bonded with no special surface preparation resulted in seams which could be manually peeled apart with minimal effort. Seams made with samples that had been wiped with xylene were also of poor quality and could readily be peeled apart. Examination of peeled specimens that had been seamed with both methods of surface preparation showed clearly that no fusion of the bonded sheets had occurred. In other words, though the two sheets had obviously melted against each other, no actual mixing of the two sheets had occurred and, on peeling, separation of the specimens occurred at the interface between the two sheets.

The surface preparation method that included abrasion of the surface followed by wiping with xylene yielded by far the best bonds. They had high tensile strength in shear and could be peeled apart only after prolonged effort by hand. They did, however, ultimately fail at the interface between the two sheets and were not equivalent to bonds made using commercially available equipment capable of mixing the two molten liner layers and thus eliminating the interface. The abraded layers adhered well because the adhering interface was of greater surface area than that of the unsanded sheets and quite irregular. The interface, however, did remain following bonding and clearly defined the zone in which bond failure had occurred.

Exposure of Heat-Seams in Test Liquids

In order to assess the effects of waste exposure on the seams, it was necessary that the seaming procedure be carried out as consistently as possible by making test seams of reproducible overlap width and seam length. The heat gun was permitted to warm-up at least 5 minutes from a cold start, and used at a constant heat setting. All seams were heated for a constant length of time and then pressed together with a roller. Elimination of interface by some means of mechanical mixing seems to make a major contribution to the peel strength of the resultant bond. The polymer chains are of high molecular weight and their ability to diffuse or move is so limited, even in the molten state, that the mixing of the two bonded layers does not seem to occur. Surface preparation definitely improves the bond strength, but it cannot eliminate this interface and so the possibility of a peel mode failure in service remains.

Thirty-two samples measuring 6.5 x 9 in. were prepared with a seam running through the middle of the sample parallel to the 6.5-in. edge using Liner No. 268, a 40-mil HDPE sheeting. These samples were used for exposing seams for shear testing. Also prepared were 32 "butterfly"-type samples measuring approximately 6.5 x 5.5 in. with a seam running along one edge, parallel to the 6.5-in. edge. These samples were used for exposing seams for peel testing. This sample size allowed five test specimens to be died out per sample. For both types of sample the grain direction of the sheeting was perpendicular to the 6.5-in. edge, i.e., perpendicular to the seam. Seaming was performed using a Leister-Kombi Type "Triac" heat gun. The samples were buffed with sandpaper and then wiped with xylene prior to seaming.

One sample was tested in shear mode for control purposes. An ASTM D638 Type I die was used to cut out specimens and five from each sample were tested. Testing was performed in accordance with ASTM D882-83, modified for seam testing in shear.

Six samples of each type of seam were placed in 1-gal of each of the following test liquids:

- Deionized water.

- 10% aqueous solution of HCl.
- 10% aqueous solution of NaOH.
- 5% mineral oil in xylene.
- Chloroform:methyl ethyl ketone:cyclohexane; 1:1:1.

The samples were removed and tested in shear at appropriate intervals. Initial test intervals were 1, 2, and 4 weeks of exposure. The remaining samples were tested after approximately 2.5 years. Test specimens were died out with a D638, Type I die. Five specimens were tested per sample.

Of the waste exposures, only 10% HCl appeared to have an effect on the HDPE seam. A total of 70 specimens and 14 seam samples were tested; eight specimens failed through a seam break in the short-term exposure. Of these eight seam failures, six occurred following exposure to 10% HCl. The tensile values at which these failures occurred were quite high, >90 ppi, but the fact that so large a proportion of the seam failures followed this exposure may be significant.

In the long-term exposures, the original seam strength values, as measured in shear, were maintained. Peel testing was performed in accordance with ASTM D413, using 0.5-in. wide specimens and a jaw separation rate of 2 inches per minute. Testing was performed in 90° peel. The peel values were low and the overall breaks were at the interfaces between the two sheets that had been seamed; unfortunately, baseline tests were not performed in the peel mode. Data show, however, that even though the shear strength values of these seams can be high, the true adhesion values as measured in peel are low. These low peel values reflect the lack of mixing of the two molten liner layers.

Data on the effects of immersion in the various test liquids on seam strength are given in Table 10.

Spin Welding as a Method of Liner Repair

Initial studies under laboratory conditions have shown that spin welding may provide a feasible means of repairing small liner defects, e.g., punctures in FMLs. This method has an advantage over seaming by hot air gun in that surface roughening or solvent cleaning is not usually required. Removal of surface debris with a brush or cloth is sufficient. The bond is made through the fusion resulting when two rotating surfaces are rubbed together; the resulting friction and pressure serve to melt the contacting surfaces, displace any surface contamination, and assure intimate contact between the bonding surfaces. Spin welding is limited in that only relatively small circular patches can be applied and so is suitable for the repair of liner punctures, but not of long tears or seam holidays.

The technique of spin welding was successfully performed using the tool shown in Figure 2. Using a drill press, it was found that an applied pressure of 100 to 150 pounds at a rotational speed of 380 rpm would fuse a

TABLE 10. EFFECTS OF IMMERSION AT ROOM TEMPERATURE IN VARIOUS TEST LIQUIDS ON SEAM STRENGTH OF HDPE[a] SEAMS

Item	Shear strength of HDPE seams on exposure in test liquid					
	Control, air	Deionized water	10% HCl	10% NaOH	5% Mineral oil in xylene	Mixed solvents
Time of immersion, days	1	11	7	7	11	11
Specimen failures[b]/specimens tested in shear	0/5	0/5	3/5	0/5	0/5	1/5
Seam strength in shear, mean, ppi	101	93	97	96	97	103
Time of immersion, days	...	25	21	21	25	25
Specimen failures[b]/specimens tested in shear	...	0/5	3/5	0/5	0/5	1/5
Seam strength in shear, mean, ppi	...	101	95	93	100	104
Time of immersion, days	...	33	...	...	33	33
Specimen failures[b]/specimens tested in shear	...	1/5	...	...	0/5	0/5
Seam strength in shear, mean, ppi	...	104	...	...	99	93
Time of immersion, days	...	976	982	982	982	...
Specimen failures[b]/specimens tested in shear	...	3/5	2/5	0/5	...[c]	...
Seam strength in shear, mean, ppi	...	105	106	104	...	...
Specimen failures[b]/specimens tested in peel	...	5/5	5/5	5/5	5/5	...
Maximum peel strength, mean, ppi	...	13	8	18	9	...

[a]40-mil HDPE sheeting. All tests were performed in the machine direction of the sheeting. Actual thicknesses of samples ranged from 36-40 mil, with an average of 37.2 mil.

[b]Failure is defined as a break at the interface between the seamed sheets, i.e., by adhesion.

[c]Specimens delaminated by hand when removed from punch press.

3-in. diameter patch within one minute. Peel strength values ranged up to 32 ppi; however, all the seams broke at the interface between the patch and the sheet being joined. This method, however, requires that the pressure be applied against a firm flat surface.

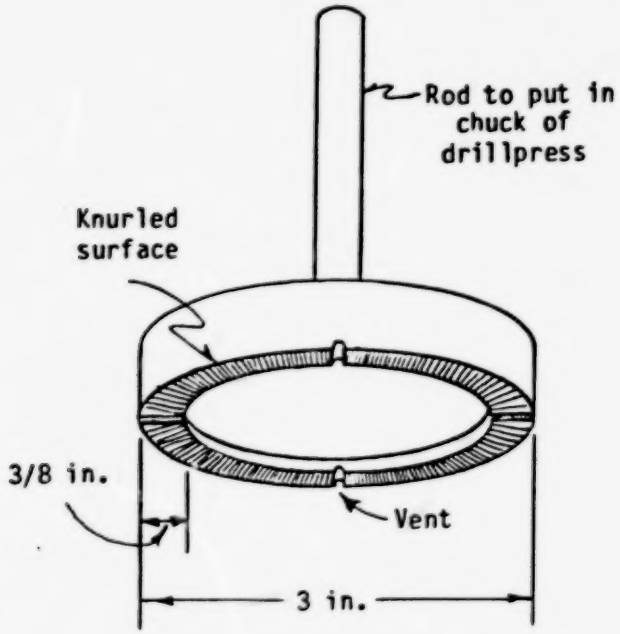


Figure 2. Schematic of a spin welding tool.

EVALUATION OF PLUGGING MATERIALS FOR REPAIRING DAMAGED LINERS

Plugging a hole with a liquid that crosslinks or hardens in place is one means of repairing a damaged liner. This procedure requires easy access to the hole and the ability to place the plugging liquid on both the upstream and downstream sides of the liner. Methods of delivering the plugging material can vary depending on the location of the hole and the types and characteristics of the liner and the plugging materials.

An hydraulic cement was used to plug a hole in an asphalt liner in a pilot study of the effects of municipal solid waste (MSW) leachate on various lining materials (Haxo et al, 1982). The leachate was drained and the column of waste above the liner was removed. A circular sample was cut from the asphalt liner, and the repair of the hole was made to resemble a sampling operation of a liner in an MSW landfill. The sample cut out exhibited a large increase in thickness during the exposure, indicating that leachate had caused the liner to swell. The repair area was prepared by digging out the gravel around the hole beneath the liner. A portion of nonmetallic, nonshrinking cement was poured into the hole, allowing an excess

of cement to seal around the edges above and below the liner to form a plug (Figure 3). The plug was allowed to harden before the liner was placed back in service in contact with MSW leachate. The cement plug made a seal that prevented leachate, which was maintained at a 1-ft hydraulic head, from passing through the hole for the remainder of the pilot study. Although the long-term effects of leachate exposure on this material are not known, the plug was quite firm when the liner was retrieved 13 months after the repair.

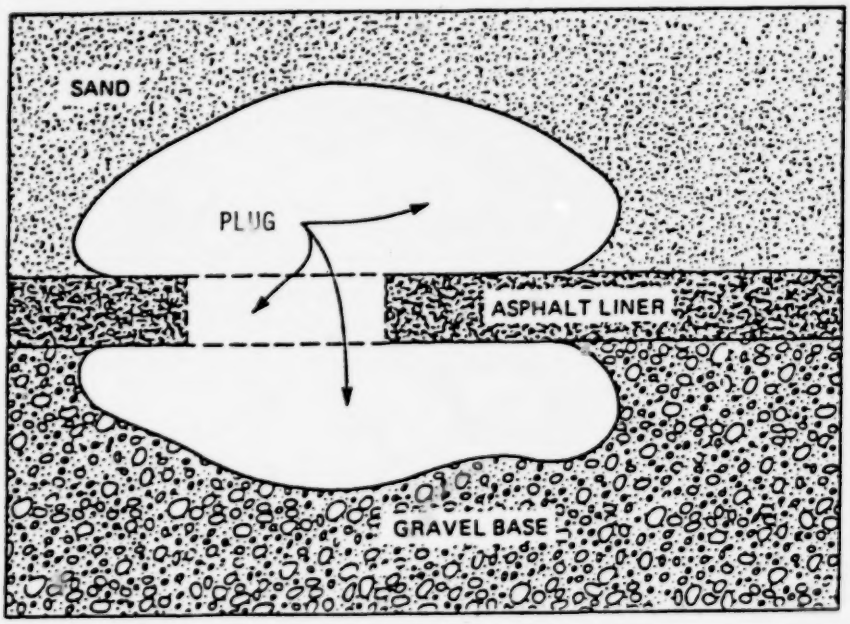


Figure 3. Plugging material made of hydraulic cement to seal hole cut in asphalt liner (Haxo et al, 1982).

The use of polymeric materials to plug leaks and holes in damaged metal drums was the subject of a previous investigation (Mitchell et al, 1973). In that project, the utility of several commercially available polymer foams was evaluated in plugging leaks in chemical containers, notably 55-gal drums. An epoxy putty and a polyurethane foam were found to seal holes in drums that contained 13 organic chemicals, but failed in drums that contained phenol, acrylonitrile, chlorosulfonic acid, and phosphorous pentasulfide. In all cases, the holes in the drums were accessible for plugging. These repairs were all intended as temporary and not as long-term repairs, which are required in the current project. Furthermore, the foams tend to have high permeability to many liquids and gases. The study also presented design concepts for foam-application equipment (Mitchell et al, 1973).

Some of the desirable characteristics of a plugging material for a permanent repair are as follows:

- Material should be curable in moist environments, even under a waste, either by moisture or by a crosslinking reaction.

- Exotherm of the crosslinking reaction should not be excessive as the heat may damage the liner.

- Material should be pumpable, have a low viscosity, and have a sufficiently long pot-life to allow delivery to the damaged area and to the hole being plugged.

- Material should be trowelable so that, if a hole is accessible, it can be repaired by hand.

- Material should be thixotropic; that is, it must be able to flow under shear and not flow when the shearing is stopped.

- Material should have a permeability comparable to that of the liner.

- Material should adhere either chemically or mechanically to the liner material. (Note: this characteristic is not necessary if the repair is temporary.)

- Material should be nonshrinking and, if possible, expandable to obtain good contact with the liner being repaired.

- Material should have a cured modulus that approximates that of the liner being repaired. Large differences in moduli between the liner and the plug may result in failure at the junction due to stress concentration.

- Material should be compatible with waste liquids and not disintegrate on prolonged exposure.

No commercial materials were located that were specifically designed to function as plugging materials for repairing lining materials used in liner systems for waste disposal facilities. On the other hand, various materials were available for repairing concrete, metals, etc., that contact water. Materials of this type were investigated for possible use as plugging materials of damaged FMLs.

The basic types of materials that appeared to be appropriate to test were:

- Epoxy resins.

- Polysulfides.

- Mixtures of epoxies and polysulfides.

- Silicones.

- Polyurethanes.

- Fast-curing inorganic cements.

- Foams.

An initial list of proprietary materials was considered. The following five epoxy materials and one fast-curing hydraulic cement were selected for testing:

- Aquatapoxy gel.

- Epocap 3331.

- Structural Adhesive 1751.

- Concresive AEX-1513.

- Sikadur Lo-Mod.

- Waterplug.

Aquatapoxy is a 2-part epoxy manufactured by American Chemical Corporation and is available in a gel mastic which is specifically designed for patching cracks and holes and can be molded and formed easily. Aquatapoxy adheres to wet, dry, or underwater surfaces and has been used to repair water settling tanks. It reportedly has good resistance to most common acids, bases, solvents, and oils. A 100-g mix has a pot-life at room temperature of approximately 40 minutes. At 70°F, tack-free cure time is 8 hours, and time for thorough cure is 36 hours.

Epocap 3331 is a 2-part epoxy multipurpose repair mastic manufactured by Hardman, Inc. This material was specifically designed for solving corrosion problems on in-water structures that are alternately exposed to air and salt water. It can be applied and cured under water and reportedly has good resistance to weathering and to alcohols, ethylene glycol, glycerine, alkalis, soaps, detergents, mineral acids in low to medium concentrations, gasoline, petroleum oils, and salt and fresh water. Pot-life of a 2-gal mix at room temperature is approximately 35 minutes. Cure time in air at 77°C is 1 to 3 days and under water is 3 to 7 days.

Scotch-weld brand Structural Adhesive 1751 is an aluminum-filled, 2-part epoxy mastic manufactured by 3M. This product is specifically designed to adhere to metal and to exhibit good void-filling properties. Pot-life of a 100-g mix at 75°F is approximately 45 minutes. Cure time in air at 60°F (i.e., until adhesive resists fingernail penetration) is approximately 16-24 hours. Parts being adhered need to be kept from moving during

cure. Contact pressure is necessary. This material was not specifically designed for use under water.

Concresive AEX-1513 is a 2-part epoxy non-sag gel manufactured by Adhesive Engineering Company. This product is specifically designed to bond precast concrete, to bond steel to concrete, and to fill gaps where resistance to chemicals is required. It reportedly has good resistance to most highly corrosive chemicals. A 225-g mix at room temperature has a pot-life of approximately 60 minutes. Tack-free cure time is approximately 4 hours, and full cure time is approximately 16 days.

Sikadur Lo-Mod is a 2-part, general purpose, epoxy adhesive manufactured by Sika Chemical Corporation. This material is marketed as a binder for broadcast aggregate and fabric-reinforced systems and is designed to adhere to concrete and steel. It is reportedly insensitive to moisture before, during, and after cure and features a low modulus which allows for "give" under impact and thermal stresses. It is not designed for application either in the presence of standing water or under water. Pot-life of a neat mix at room temperature is approximately 50 minutes. Tack-free cure time of a thin film is approximately 4 hours. Final cure (75% of ultimate strength) is reached in approximately 3 days.

Waterplug is a hydraulic cement manufactured by Thoro System Products. It is designed to seal masonry and concrete leaks and will expand and harden in place. It reportedly can be used to seal running water leaks, including leaks under water. Set time ranges from 3 to 5 minutes.

Experimental Details

The five proprietary epoxy compositions were tested as received, i.e., without any additives for improving rheological or curing characteristics. The following properties were measured for each composition:

- Gel time of a 15-g aliquot.
- Viscosity at 25°C of the mixed epoxy.
- Thixotropy, as determined by a ratio of viscosity measurements and by a slope test.
- Density, in g/cm^3, of each component and of the mixture.
- Hardness, in Duro D points, of the cured mixture.
- Compatibility with water and five different test media.

The Waterplug hydraulic cement was only tested for cure time and for thixotropy by the slope test. The results of this testing are presented in Table 11.

TABLE 11. TRADE NAMES AND PHYSICAL CHARACTERISTICS OF PLUGGING MATERIALS SELECTED FOR TEST

Property	Trade name, type, and supplier					
	Aquatapoxy, 2-part epoxy, American Chemical Corp.	Epocap 3331 2-part epoxy, Hardman, Inc.	Structural Adhesive 1751, 2-part epoxy, aluminum filled, 3M, Coatings and Sealers Division	Concresive AEX-1513, 2-part epoxy, Adhesive Engineering Company	Sikadur Lo-Mod Gel, 2-part epoxy, Sika Chemical Corporation	Waterplug, hydraulic cement, Standard Dry Wall Products
Gel time of 15-g aliquot	6 h	3 h	5 h	6 h	4 h	...
Cure time of 15-g aliquot	36 h	1 to 3 d	12 h	16 d	3 d	3 min.
Viscosity[a] at 25°C						
Poise at 2.5 rpm	4,000	19,200	8,000	10,000	500	...
Poise at 5.0 rpm	2,600	15,000	5,400	6,600	400	...
Shear index[b]	1.54	1.28	1.48	1.52	1.25	...
Slope test[c], cm	0.6 cm	0 cm	0.9 cm	0 cm	5.3 cm	>10 cm[d]
Density, g/cm^3						
Component A	1.57	1.82	0.95	1.14	1.70	...
Component B	0.96	1.40	1.30	1.10	1.59	...
Mixture of A and B[e]	1.26	1.61	1.16	1.13	1.66	...
Hardness Duro D, 5-sec.	69	58	69	53	59	...

[a]Brookfield viscosity of mixed composition using spindle T-F and Helipath.

[b]Shear index: viscosity at 2.5 rpm divided by viscosity at 5.0 rpm.

[c]Slope test: movement of 10- to 12-gram sample down a 9° slope covered with HDPE.

[d]Flowed significantly.

[e]Calculated from volume mixture ratio.

These epoxy compositions are two-part systems which were mixed either by hand or in Semkits (USAF, 1973). The Semkits obtained from Adhesive Sealants Corp., Paramount, California, were loaded with the two parts of the epoxy which were separated by a barrier. Upon rupture of the barrier, the two parts were mixed with the plunger in the Semkit.

Viscosity of the mixed epoxies was measured at 25°C in accordance with ASTM D2393 at two shear rates, 2.5 rpm and 5.0 rpm, with a Brookfield viscometer using the T-F spindle and Helipath.

In order to be placed satisfactorily, a plugging material should possess a degree of thixotropy; that is, while the material must be readily pumpable or injectable, it must also have enough body to resist gradual flow or creep while hardening. Two tests, the determination of a shear index and the measurement of flow on a slope, were used to evaluate this property. The results of the viscosity measurements at two shear rates (2.5 and 5.0 rpm), determined in accordance with ASTM D2393, were used to calculate the shear index of each material. In the slope test, a small incline covered with HDPE sheeting and having a 9° slope was used. One ounce of material was poured into a form, the form removed, and the length of flow down the slope was measured. These tests were conducted at room temperature.

Specimens for tests of hardness and compatibility with various test liquids were prepared by pouring 15-g aliquots of a material into aluminum foil dishes and leveling the masses with a Teflon trowel to yield disks 6 cm in diameter and 0.5 to 0.6 cm in depth. The tendency of each of these materials to absorb and/or degrade following exposure to waste was evaluated by monitoring changes in weight and hardness on immersion of the specimens in various test liquids. The test liquids selected for these compatibility tests were:

- Deionized water.
- 10% HCl.
- 10% NaOH.
- 5% mineral oil in xylene.
- Solvent mixture of chloroform:methyl ethyl ketone: cyclohexane; 1:1:1.

These liquids were selected to simulate extreme conditions of chemical concentrations.

The compatibility tests were performed in accordance with two different procedures. In the first procedure, the ability of the plugging materials to harden and cure in situ was assessed. The test specimens were immersed in the test media immediately after mixing. In the second procedure, the specimens were allowed to cure for one day at room temperature before they were immersed in the test media. All immersions were at room temperature. In both procedures, the weight and durometer changes were determined after

various immersion times. Hardness was measured in accordance with ASTM D2240 using either a Shore A or Shore D durometer, depending on the hardness of the disk specimens. The Waterplug hydraulic cement was only tested in accordance with the second procedure, and the effects of the immersions were only monitored by changes in weight.

Compatibility was thus evaluated both during *in situ* cure and after cure. In each case, a sample aliquot cured in air at room temperature was used as a control. The test results for the 6 plugging materials in both exposure tests are reported for 1, 2, 3, and 4 weeks of immersion in Tables 12 through 16. As all of the individual measurements of weight and hardness could not be made exactly on weekly intervals, results include interpolated and extrapolated data which are so indicated in these tables. The results for 4 weeks of immersion are summarized in Table 17.

Conclusions Regarding Plugging

Plugging appears to be a promising method for making *in situ* repairs of accessible holes in FMLs that are in-service if adhesion to the liner is not required. However, the compatibility of a potential plugging material with the waste liquid to be contained should be assessed to determine:

- Whether the plugging material can harden under the waste.
- If the plugging material does harden under the waste, whether compatibility with the waste liquid will be maintained for extended periods of time.
- If the plugging material is allowed to harden in air, whether it can withstand long-term exposure to the waste liquid.

All of the plugging materials tested in this project appeared to harden in the presence of water; however, none cured in the presence of the mixed solvents, and only the Waterplug appeared to withstand exposure to this test medium. The highly acidic environment inhibited the curing of all the materials except the Aquatapoxy. The alkaline environment inhibited the curing of three of the five epoxies, i.e., Epocap 3331, Structural Adhesive 1751, and the Sikadur Lo-Mod; the Aquatapoxy and the Concressive AEX-1513 cured. The responses of the five epoxies to the 5% mineral oil in xylene were similar. Generally, if the epoxy is allowed to cure before being exposed it will hold up better than if it cures *in situ*.

The epoxy systems were characterized by high viscosity and thixotropy, two properties which enable the plug to resist displacement while curing. The epoxies were also chosen to have long gel times to maximize the amount of time over which the material could be worked and applied.

The hydraulic cement initially formed a liquid the consistency of heavy cream, setting within three minutes to a hard mass. Its properties were significantly different than those of any of the epoxies we tested and would require a totally different application technique than that used with 2-part epoxies.

TABLE 12. HARDNESS AND PERCENT WEIGHT CHANGE ON IMMERSION OF PLUGGING MATERIALS IN DEIONIZED WATER

	Plugging material											
	Aquatapoxy		Epocap 3331		Structural Adhesive 1751		Concresive AEX-1513		Sikadur Lo-Mod		Waterplug	
Time/weeks	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %
Control[c]	69D	0	66D	0	69D	0	53D	0	59D	0	...	0
Cured under deionized water												
1	52D[d]	8.0	51D	2.6	36A	16.2	62D	2.9	34D	10.0	...	...
2	52D	1.9	55D	7.0	44A	12.9	67D	1.4	44D	4.0	...	...
3	53D	1.4	61D	2.8	48A	10.2	66D	1.7	45D	5.4	...	...
4	52D	0.9	65D	2.7	48A	14.6	56D	1.7	40D	4.2	...	...
Cured in air for one day and then immersed in deionized water												
1	60D	1.3	~60A	10.5	53D	1.5	55D	3.0	56D	3.4	...	-6.0
2	60D	2.0	36A	11.0	48D	1.0	55D	3.9	55D	4.6	...	-8.2
3	66D	1.6	31A	15.0	50D	1.0	54D	1.6	55D	3.2	...	-7.7
4	64D	0.3	37A	16.6	51D	1.1	55D	1.6	57D	3.0	...	-8.0

[a]5-second durometer hardness readings; durometer scale indicated by letter following value.

[b]Percent change in weight of disk.

[c]Ultimate hardness reached on cure at room temperature.

[d]Underlined data are interpolated or extrapolated.

TABLE 13. HARDNESS AND PERCENT WEIGHT CHANGE ON IMMERSION OF PLUGGING MATERIALS IN 10% HCl

	Plugging material											
	Aquatapoxy		Epocap 3331		Structural Adhesive 1751		Concresive AEX-1513		Sikadur Lo-Mod		Waterplug	
Time/weeks	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %
Control[c]	69D	0	66D	0	69D	0	53D	0	59D	0	...	0
Cured under 10% HCl												
1	54D[d]	2.4	44A	9.5	40D	-1.0	...	-23.0	...	4.9	...	...
2	57D	1.3	45A	13.4	36D	0.7	...	<-23.0	...	7.6	...	...
3	59D	...	45A	14.0	40D	0.7	...	...	...	8.6	...	...
4	59D	...	45A	16.2	60A	0.8	...	...	...	6.8	...	...
Cured in air for one day and then immersed in 10% HCl												
1	50D	3.8	30A	40.5	45D	9.3	58D	3.5	64D	3.4	...	-81.4
2	59D	4.3	36A	53.0	38D	13.7	53D	3.0	58D	3.9	...	ca -85.0
3	62D	4.5	46A	59.0	31D	13.0	57D	...	54D	4.6	...	...
4	48D	4.7	30A	60.0	37D	14.0	58D	...	53D	5.0	...	...

[a]5-second durometer hardness readings; durometer scale indicated by letter following value.

[b]Percent change in weight of disk.

[c]Ultimate hardness reached on cure at room temperature.

[d]Underlined data are interpolated or extrapolated.

TABLE 14. HARDNESS AND PERCENT WEIGHT CHANGE ON IMMERSION OF PLUGGING MATERIALS IN 10% NaOH

	Plugging material											
	Aquatapoxy		Epocap 3331		Structural Adhesive 1751		Concresive AEX-1513		Sikadur Lo-Mod		Waterplug	
Time/weeks	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %
Control[c]	69D	0	66D	0	69D	0	53D	0	59D	0	...	0
Cured under 10% NaOH												
1	30D[d]	12.8	33D	4.1	...	4.4	50D	3.2	...	2.6	...	...
2	29D	10.1	33D	5.4	...	1.2	51D	17.0	...	3.5	...	...
3	30D	10.4	70A	6.8	...	1.8	46D	17.2	...	0.8	...	...
4	33D	10.0	60A	7.7	...	2.6	49D	17.4	...	4.0	...	...
Cured in air for one day and then immersed in 10% NaOH												
1	70D	2.7	60A	6.7	43D	6.4	75D	2.0	55D	3.6	...	-2.2
2	75D	3.2	50A	8.5	39D	7.0	63D	2.0	52D	4.0	...	-1.8
3	59D	2.2	32A	11.0	50D	6.4	65D	2.0	54D	3.7	...	-3.1
4	61D	2.2	38A	12.0	52D	>6.0	63D	2.0	53D	3.9	...	...

[a]5-second durometer hardness readings; durometer scale indicated by letter following value.

[b]Percent change in weight of disk.

[c]Ultimate hardness reached on cure at room temperature.

[d]Underlined data are interpolated or extrapolated.

TABLE 15. HARDNESS AND PERCENT WEIGHT CHANGE ON IMMERSION OF PLUGGING MATERIALS IN 5% MINERAL OIL IN XYLENE

	Plugging material											
	Aquatapoxy		Epocap 3331		Structural Adhesive 1751		Concresive AEX-1513		Sikadur Lo-Mod		Waterplug	
Time/weeks	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %
Control[c]	69D	0	66D	0	69D	0	53D	0	59D	0	...	0
Cured under 5% mineral oil in xylene												
1	soft	>12.0[d]	42D	20.5	37A	39.8	42D	9.8	soft	60.5	...	...
2	...	...	60A	22.8	37A	43.0	37D	10.8	...	59.1	...	...
3	...	...	55A	22.5	36A	44.0	27D	10.8	...	58.0	...	...
4	...	...	55A	22.2	38A	43.1	38D	>12.0	soft	>58.0	...	...
Cured in air for one day and then immersed in 5% mineral oil in xylene												
1	60D	31.1	40D	8.2	60A	22.2	69D	3.1	25D	27.3	...	-1.3
2	50D	29.0	34D	12.0	50A	32.0	45D	4.6	63A	>45.0	...	-0.8
3	22D	27.3	36D	13.8	51A	36.0	40D	5.8	56A	>45.0	...	-1.3
4	19D[e]	27.4	80A	15.7	55A	37.5	40D	7.1	51A	>45.0	...	...

[a]5-second durometer hardness readings; durometer scale indicated by letter following value.

[b]Percent change in weight of disk.

[c]Ultimate hardness reached on cure at room temperature.

[d]Underlined data are interpolated or extrapolated.

[e]Continued to soften after 28 days.

TABLE 16. HARDNESS AND PERCENT WEIGHT CHANGE OF IMMERSION OF PLUGGING MATERIALS IN A SOLVENT MIXTURE OF CHLOROFORM:METHYL ETHYL KETONE:CYCLOHEXANE 1:1:1

	Plugging material											
	Aquatapoxy		Epocap 3331		Structural Adhesive 1751		Concresive AEX-1513		Sikdaur Lo-Mod		Waterplug	
Time/weeks	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %	Duro[a]	Δwt[b], %
Control[c]	69D	0	66D	0	69D	0	53D	0	59D	0	...	0
Cured under mixed solvents												
1	did not harden	...	...	9.8	10A[d]	>53.0	36A	-13.6	...	...	...	...
2	...	...	...	...	10A	...	35A	...	...	...	...	...
3	...	...	...	...	10A	...	35A	...	...	...	...	...
4	...	...	...	...	10A	...	37A	...	...	...	...	...
Cured in air for one day and then immersed in mixed solvents												
1	soft and sticky	54.4	soft	40.3	soft	107.6	24D	21.0	58A	81.2	...	-5.4
2	...	>50.0	...	45.0	...	108.0	24D	28.4	50A	65.0	...	-5.4
3	...	>50.0	...	40.0	...	110.0	25D	30.0	48A	55.0	...	-5.1
4	...	...	...	...	...	...	65A	31.0	50A	...	...	...

[a]5-second durometer hardness readings; durometer scale indicated by letter following value.

[b]Percent change in weight of disk.

[c]Ultimate hardness reached on cure at room temperature.

[d]Underlined data are interpolated or extrapolated.

TABLE 17. SUMMARY[a] OF IMMERSION TEST IN VARIOUS LIQUID MEDIA FOR FOUR WEEKS

Hardness and Percent Weight Change of Potential Plugging Materials

Test medium	Cure	Plugging material: Aquatapoxy		Epocap 3331		Structural Adhesive 1751		Concresive AEX-1513		Sikadur Lo-Mod		Waterplug	
		Duro[b]	Δwt[c], %	Duro[b]	Δwt[c], %	Duro[b]	Δwt[c], %	Duro[b]	Δwt[c], %	Duro[b]	Δwt[c], %	Duro[b]	Δwt[c], %
Control[d]		69D	0	66D	0	69D	0	58D	0	59D	0	...	0
DI H_2O	In situ[e]	52D	0.9	61D	2.7	48A	14.6	56D	1.7	40D	4.2	...	...
DI H_2O	Air[f]	64D	0.3	37A	16.6	51D	1.1	55D	1.6	57D	3.0	...	-8.0
10% HCl	In situ[e]	59D	...	45A	16.2	60A	0.8	...	...	...	6.8	...	...
10% HCl	Air[f]	48D	4.7	30A	60.0	37D	14.0	58D	...	53D	5.0	...	<-85
10% NaOH	In situ[e]	33D	10.0	60A	7.7	...	2.6	49D	17.4	...	4.0	...	...
10% NaOH	Air[f]	61D	2.2	38A	12.0	52D	>6.0	63D	2.0	53D	3.9	...	<-3.1
5% mineral oil in xylene	In situ[e]	52D	0.9	55A	22.2	38A	43.1	38D	>12.0	soft	>58.0	...	...
	Air[f]	19D	27.4	80A	15.7	55A	37.5	40D	7.1	51A	>45.0	...	<-1.3
Solvent mixture[g]	In situ[e]	soft	...	soft	...	10A	>53.0	37A	...	...	...	...	...
	Air[f]	soft	>50	...	...	...	...	65A	31.0	50A	...	...	ca -5.0

[a]From Tables 11 through 16.

[b]5-second durometer hardness readings; durometer scale indicated by letter following value.

[c]Percent change in weight of disk.

[d]Ultimate hardness reached on cure at room temperature.

[e]15 g mass of plugging material in aluminum dish placed immediately into test medium to cure.

[f]Specimen allowed to cure one day at room temperature in air before being immersed in test liquid.

[g]Solvent mixture: chloroform:methyl ethyl ketone:cyclohexane; 1:1:1.

Overall, it is quite apparent that long-term compatibility tests must be performed on candidate plugging materials before they can be depended on for "permanent" repairs.

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